Banks, Kendra 219064

From: JOHN CHU [john.chu@uspto.gov]
Sent: Wednesday, March 21, 2007 7:54 PM
To: STIC-EIC1700
Subject: Database Search Request, Serial Number: 10773,366

Requester:
JOHN CHU (P/1752)
Art Unit:
GROUP ART UNIT 1752
Employee Number:
68314
Office Location:
REM 09D51
Phone Number:
(571)272-1329
Mailbox Number:

Case serial number:
10773,366
Class / Subclass(es):
430/157
Earliest Priority Filing Date:

Format preferred for results:
Paper
Search Topic Information:
Please search claim 19 for the azolinyl acetic acid derivative and then its use as a coupler in a heat sensitive recording material.

As background of the chemistry: The diazonium compound couples with the compound of claim 19 upon heating to give an azo dye, thus forming a color.

Thanks you!!

John
Special Instructions and Other Comments:

```
=> FILE REG
FILE 'REGISTRY' ENTERED AT 18:07:28 ON 28 MAR 2007
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
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=> D HIS

     FILE 'LREGISTRY' ENTERED AT 17:47:47 ON 28 MAR 2007
L1              STR

     FILE 'REGISTRY' ENTERED AT 17:52:32 ON 28 MAR 2007
L2            40 S L1
L3           659 S L1 FUL
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L4              STR L1

     FILE 'REGISTRY' ENTERED AT 17:58:31 ON 28 MAR 2007
L5             1 S L4 SSS SAM SUB=L3
L6            60 S L4 SSS FUL SUB=L3
                 SAV L6 CHU366A/A

     FILE 'CAOLD' ENTERED AT 17:59:21 ON 28 MAR 2007
L7             0 S L6
L8            18 S L3
L9          6884 S ?DIAZO?
L10            0 S L8 AND L9

     FILE 'HCA' ENTERED AT 17:59:58 ON 28 MAR 2007
L11           17 S L6
L12          403 S L3
L13              QUE ?DIAZO?
L14           58 S L12 AND L13
L15       751486 S COUPL?
L16           22 S L14 AND L15
L17            9 S L11 AND L16
L18           17 S L17 OR L11
L19           13 S L16 NOT L18
L20       377755 S RECORD?
L21        39927 S (HEAT? OR THERMAL? OR THERMO?)(2A)SENS?
L22           10 S L14 AND L20
L23            2 S L14 AND L21
L24           13 S (L16 OR L22 OR L23) NOT L18
L25           16 S 1840-2004/PY,PRY AND L18
L26           13 S 1840-2004/PY,PRY AND L24
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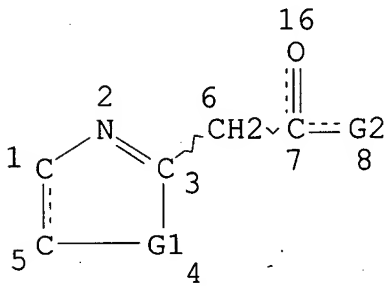
FILE 'REGISTRY' ENTERED AT 18:07:28 ON 28 MAR 2007

=> D L6 QUE STAT
L1 STR



C @11

VAR G1=O/S
VAR G2=11/N/O
NODE ATTRIBUTES:
NSPEC IS RC AT 11
DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
RING(S) ARE ISOLATED OR EMBEDDED
NUMBER OF NODES IS 10

STEREO ATTRIBUTES: NONE
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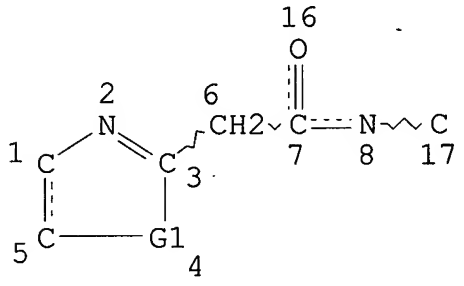
L4 STR



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DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
RSPEC I

NUMBER OF NODES IS 10

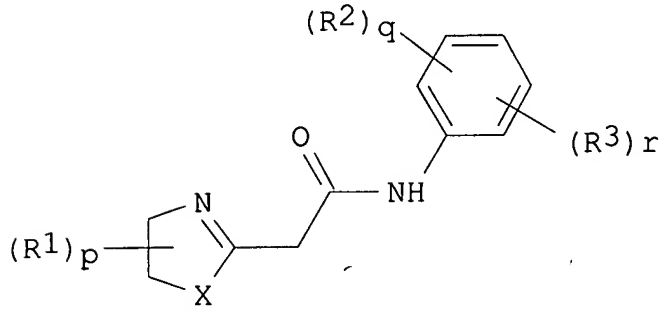
STEREO ATTRIBUTES: NONE
L6 60 SEA FILE=REGISTRY SUB=L3 SSS FUL L4

100.0% PROCESSED 331 ITERATIONS 60 ANSWERS
SEARCH TIME: 00.00.01

=> FILE HCA
FILE 'HCA' ENTERED AT 18:07:50 ON 28 MAR 2007

=> D L25 1-16 CBIB ABS HITSTR HITIND

L25 ANSWER 1 OF 16 HCA COPYRIGHT 2007 ACS on STN
143:430092 Heterocyclic **coupler** compound and its use together with microencapsulated **diazo** compound in **diazo** recording paper. Higuchi, Satoshi; Ikeda, Takayoshi (Fuji Photo Film Co., Ltd., Japan). Jpn. Kokai Tokkyo Koho JP 2005298446 A 20051027, 44 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 2004-120467 20040415.
GI

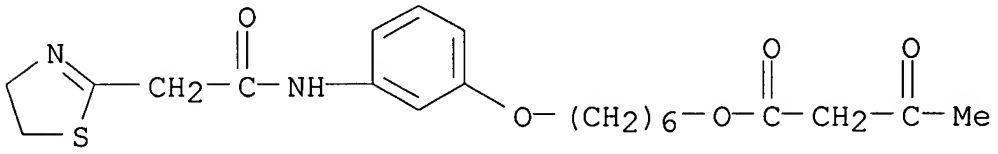


I

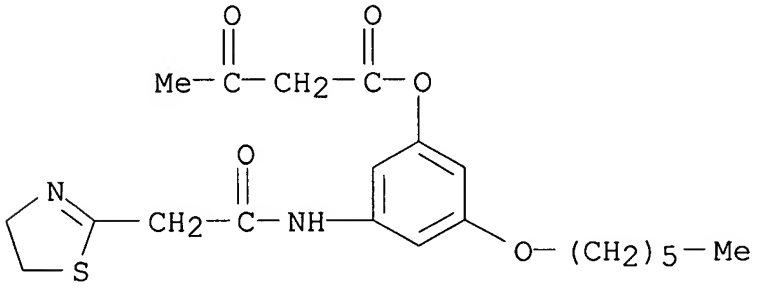
AB The title heterocyclic **coupler** compd. is represented by a general formula I (X = O, S; R1, R2 = substituent; R3 = substituent having 1,3-dicarbonyl structure; p, q = 0-4; r = 1-5). 2 Synthetic examples and 4 **diazo** recording paper examples are given.

IT **868272-22-4 868272-23-5**
(heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)

RN 868272-22-4 HCA
CN Butanoic acid, 3-oxo-, 6-[3-[[(4,5-dihydro-2-thiazolyl)acetyl]amino]phenoxy]hexyl ester (9CI) (CA INDEX NAME)

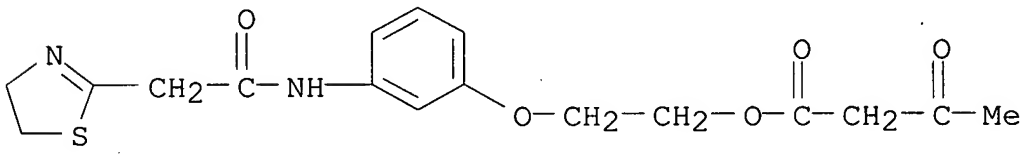


RN 868272-23-5 HCA
CN Butanoic acid, 3-oxo-, 3-[[(4,5-dihydro-2-thiazolyl)acetyl]amino]-5-(hexyloxy)phenyl ester (9CI) (CA INDEX NAME)



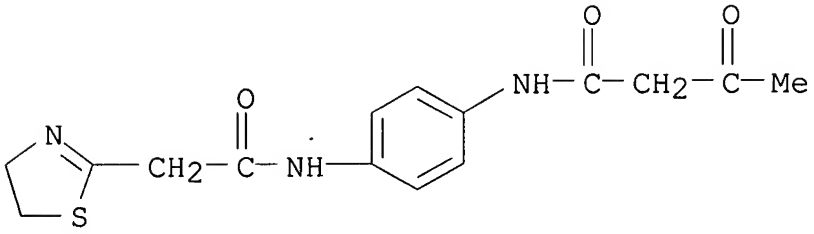
IT **868272-18-8P 868272-21-3P**
(prepn. of heterocyclic **coupler** compd.; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)

RN 868272-18-8 HCA
CN Butanoic acid, 3-oxo-, 2-[3-[[(4,5-dihydro-2-thiazolyl)acetyl]amino]phenoxy]ethyl ester (9CI) (CA INDEX NAME)



RN 868272-21-3 HCA
CN 2-Thiazoleacetamide, N-[4-[(1,3-dioxobutyl)amino]phenyl]-4,5-dihydro-

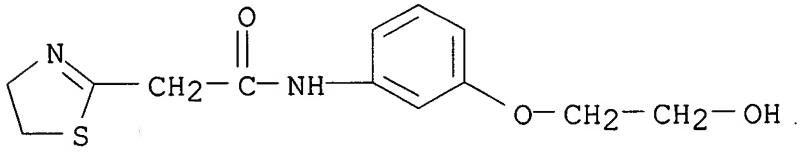
(9CI) (CA INDEX NAME)



IT **868272-17-7P 868272-19-9P 868272-20-2P**
(prepn. of heterocyclic **coupler** compd.; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)

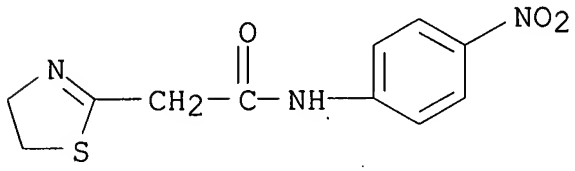
RN 868272-17-7 HCA

CN 2-Thiazoleacetamide, 4,5-dihydro-N-[3-(2-hydroxyethoxy)phenyl]- (9CI) (CA INDEX NAME)



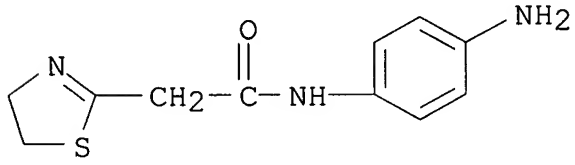
RN 868272-19-9 HCA

CN 2-Thiazoleacetamide, 4,5-dihydro-N-(4-nitrophenyl)- (9CI) (CA INDEX NAME)



RN 868272-20-2 HCA

CN 2-Thiazoleacetamide, N-(4-aminophenyl)-4,5-dihydro- (9CI) (CA INDEX NAME)



IC ICM C07D277-10
ICS B41M005-28; B41M005-30
CC 74-10 (Radiation Chemistry, Photochemistry, and Photographic and Other Reprographic Processes)
ST heterocyclic **coupler** microencapsulated **diazo** compd recording paper
IT Copying paper
(**diazo**; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)
IT Polyurethanes, uses
(microcapsule; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)
IT **Diazo** process
(paper; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)
IT 663934-47-2
(**diazo** compd.; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)
IT **868272-22-4 868272-23-5**
(heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)
IT 148130-89-6
(microcapsule; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)
IT **868272-18-8P 868272-21-3P**
(prepn. of heterocyclic **coupler** compd.; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)
IT 60-23-1, 2-Aminoethanethiol 100-01-6, 4-Nitroaniline, reactions 372-09-8, Cyano acetic acid 674-82-8, Diketene 16365-26-7, 2-(3-Nitrophenoxy)-ethanol
(prepn. of heterocyclic **coupler** compd.; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)
IT 22208-39-5P 50963-77-4P 128259-54-1P **868272-17-7P 868272-19-9P 868272-20-2P**
(prepn. of heterocyclic **coupler** compd.; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo**

recording paper)

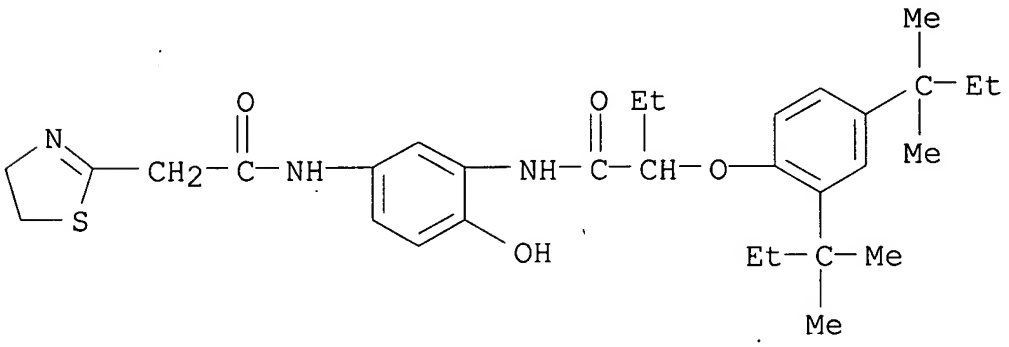
L25 ANSWER 2 OF 16 HCA COPYRIGHT 2007 ACS on STN
143:430091 Heterocyclic **coupler** compound and its use together with microencapsulated **diazo** compound in **diazo** recording paper. Higuchi, Satoshi; Ikeda, Takayoshi (Fuji Photo Film Co., Ltd., Japan). Jpn. Kokai Tokkyo Koho JP 2005298445 A 20051027, 45 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 2004-120466 20040415.
GI

* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB The title heterocyclic **coupler** compd. is represented by I or II (X = O, S; R1, R2 = substituent; R3 = acyl, carbamoyl, alkoxycarbonyl, alkylsulfonyl, arylsulfonyl; m = 0-4; n = 0-3; o, p = 0, 1). 4 Synthetic examples and 4 **diazo** recording paper examples are given.

IT **868274-09-3**
(heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)

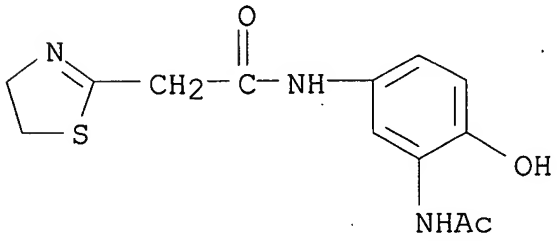
RN 868274-09-3 HCA
CN 2-Thiazoleacetamide, N-[3-[[2-[2,4-bis(1,1-dimethylpropyl)phenoxy]-1-oxobutyl]amino]-4-hydroxyphenyl]-4,5-dihydro- (9CI) (CA INDEX NAME)



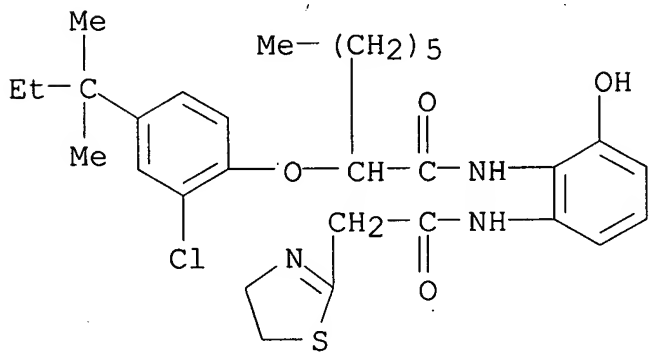
IT **868274-02-6P 868274-04-8P 868274-06-0P 868274-08-2P**
(prepn. of heterocyclic **coupler** compd.; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)

RN 868274-02-6 HCA

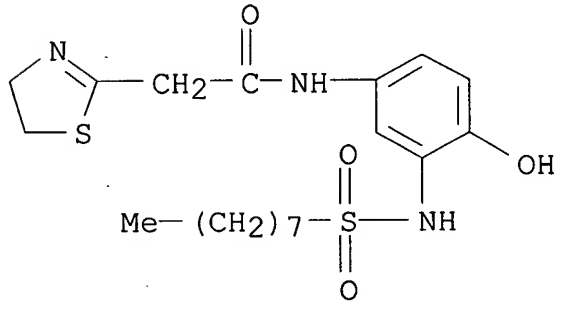
CN 2-Thiazoleacetamide, N-[3-(acetylamino)-4-hydroxyphenyl]-4,5-dihydro- (9CI) (CA INDEX NAME)



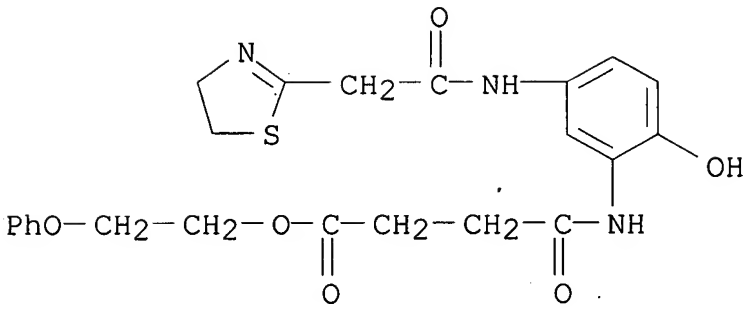
RN 868274-04-8 HCA
CN 2-Thiazoleacetamide, N-[2-[[2-[2-chloro-4-(1,1-dimethylpropyl)phenoxy]-1-oxooctyl]amino]-3-hydroxyphenyl]-4,5-dihydro- (9CI) (CA INDEX NAME)



RN 868274-06-0 HCA
CN 2-Thiazoleacetamide, 4,5-dihydro-N-[4-hydroxy-3-[(octylsulfonyl)amino]phenyl]- (9CI) (CA INDEX NAME)



RN 868274-08-2 HCA
CN Butanoic acid, 4-[[5-[[(4,5-dihydro-2-thiazolyl)acetyl]amino]-2-hydroxyphenyl]amino]-4-oxo-, 2-phenoxyethyl ester (9CI) (CA INDEX NAME)



IC ICM C07D277-10
ICS B41M005-124; B41M005-165; B41M005-28; B41M005-30
CC 74-10 (Radiation Chemistry, Photochemistry, and Photographic and Other Reprographic Processes)
ST heterocyclic **coupler** microencapsulated **diazo** compd recording paper
IT Copying paper
(**diazo**; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)
IT Polyurethanes, uses
(microcapsule; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)
IT **Diazo** process
(paper; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)
IT 663934-47-2
(**diazo** compd.; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)
IT **868274-09-3**
(heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)
IT 148130-89-6
(microcapsule; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)
IT **868274-02-6P 868274-04-8P 868274-06-0P 868274-08-2P**
(prepn. of heterocyclic **coupler** compd.; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)

IT 60-23-1, 2-Aminoethanethiol 99-57-0, 2-Amino-4-nitrophenol
372-09-8, Cyanoacetic acid 868274-03-7 868274-05-9 868274-07-1
(prepn. of heterocyclic **coupler** compd.; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)

IT 64-19-7P, Acetic acid, reactions 97-60-9P 23184-60-3P
868274-01-5P
(prepn. of heterocyclic **coupler** compd.; heterocyclic **coupler** compd. and its use together with microencapsulated **diazo** compd. in **diazo** recording paper)

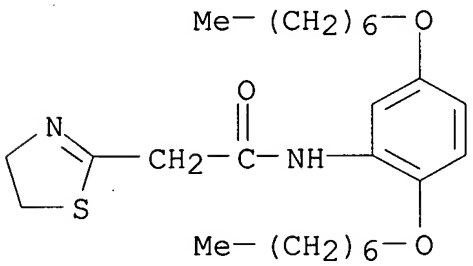
L25 ANSWER 3 OF 16 HCA COPYRIGHT 2007 ACS on STN
143:413561 Organic base content-controlled heat-developable **diazo** recording material with. Ikeda, Kimi (Fuji Photo Film Co., Ltd., Japan). Jpn. Kokai Tokkyo Koho JP 2005297378 A 20051027, 69 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 2004-117538 20040413.

AB The material has a recording layer contg. a **diazo** compd., a **coupler**, and an org. base with pka >5 in less mol content than that of the **diazo** compd. The material shows improved color development, hue, and less light stain.

IT **769171-75-7**
(**coupler**; org. base content-controlled heat-developable **diazo** recording material)

RN 769171-75-7 HCA

CN 2-Thiazoleacetamide, N-[2,5-bis(heptyloxy)phenyl]-4,5-dihydro- (9CI) (CA INDEX NAME)



IC ICM B41M005-30

CC 74-10 (Radiation Chemistry, Photochemistry, and Photographic and Other Reprographic Processes)

ST heat developable **diazo** recording material org base content; enamine structure **coupler diazo** recording material

IT **Diazo** process
(heat-developable; org. base content-controlled heat-developable

diazo recording material)

IT **769171-75-7** 867380-72-1 867380-73-2
(**coupler**; org. base content-controlled heat-developable **diazo** recording material)

IT 67928-21-6 663934-47-2
(**diazonium** salt; org. base content-controlled heat-developable **diazo** recording material)

IT 101-01-9
(org. base content-controlled heat-developable **diazo** recording material)

L25 ANSWER 4 OF 16 HCA COPYRIGHT 2007 ACS on STN

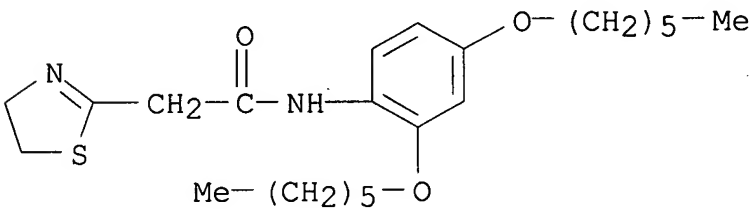
143:356712 Heat-developable **diazo** recording material containing protonated dye. Yanagihara, Naoto; Ikeda, Kimi; Takeuchi, Yosuke (Fuji Photo Film Co., Ltd., Japan). Jpn. Kokai Tokkyo Koho JP 2005271301 A 20051006, 53 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 2004-85307 20040323.

AB The material comprises a support coated with a heat-sensitive layer contg. protonated dye formed by the reaction of a **diazonium** salt and a **coupler**. The material shows good lightfastness.

IT **865619-36-9**
(**coupler**, for yellow dye formation; heat-developable **diazo** recording material contg. protonated dye for lightfastness)

RN 865619-36-9 HCA

CN 2-Thiazoleacetamide, N-[2,4-bis(hexyloxy)phenyl]-4,5-dihydro- (9CI) (CA INDEX NAME)



IC ICM B41M005-30
ICS B41M005-26

CC 74-10 (Radiation Chemistry, Photochemistry, and Photographic and Other Reprographic Processes)

ST heat developable **diazo** recording material lightfastness; protonated yellow dye **diazonium** salt **coupler**

IT Microcapsules
(contg. **diazonium** salt; heat-developable **diazo** recording material contg. protonated dye for lightfastness)

IT **Diazo** process
(heat-developable; heat-developable **diazo** recording

material contg. protonated dye for lightfastness)

IT **865619-36-9** 865619-37-0
(**coupler**, for yellow dye formation; heat-developable **diazo** recording material contg. protonated dye for lightfastness)

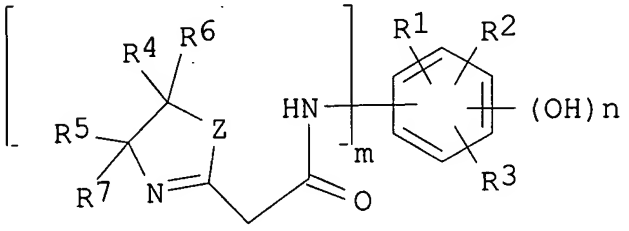
IT 663934-47-2
(**diazonium** salt, yellow; heat-developable **diazo** recording material contg. protonated dye for lightfastness)

IT 77-99-6D, Trimethylolpropane, reaction products with xylylene diisocyanate 80-05-7D, Bisphenol A, reaction products with xylylene diisocyanate 25854-16-4D, Xylylene diisocyanate, reaction products with trimethylolpropane and/or bisphenol A 266309-16-4, Takenate D 119N
(microcapsule from; heat-developable **diazo** recording material contg. protonated dye for lightfastness)

IT 865619-38-1
(photoacid generator; heat-developable **diazo** recording material contg. protonated dye for lightfastness)

L25 ANSWER 5 OF 16 HCA COPYRIGHT 2007 ACS on STN
142:326011 Thermal recording material containing **diazo** compound and anilide derivative **coupler**. Arioka, Daisuke; Ikeda, Takayoshi (Fuji Photo Film Co., Ltd., Japan). Jpn. Kokai Tokkyo Koho JP 2005074948 A 20050324, 42 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 2003-311431 20030903.

GI



I

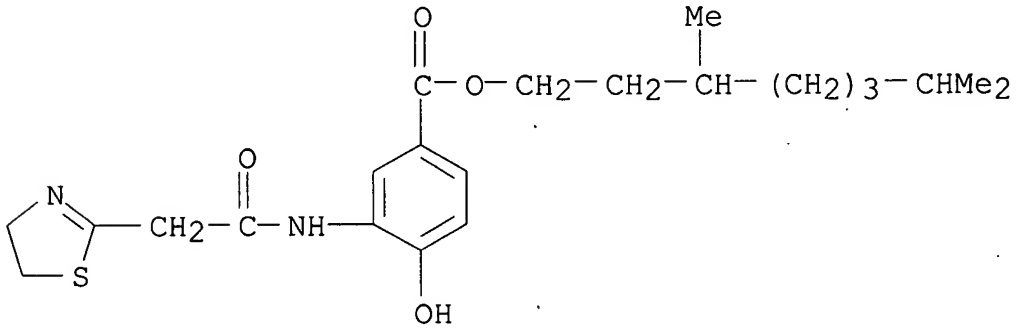
AB The material has a heat-sensitive layer contg. the **diazo** compd. and the **coupler** contg. an anilide deriv. I (R1-3 = H, halo, alkyl, aryl, acyl, alkoxy, aryloxy, alkoxycarbonyl, carboxyl, aminocarbonyl, acylamino, aminosulfonyl, sulfonamide, CN, NO2, arylthio, alkylthio; R4-7 = H, alkyl, aryl, alkoxycarbonyl, amide; Z = S, O, NR8; R8 = H, alkyl, aryl; m = 1, 2; n = 1, 2) and/or its tautomer. The material shows improved raw-stock stability and image stability to light.

IT **848251-94-5P**
(**coupler**; heat-developable **diazo** recording

material contg. anilide deriv. **coupler**)

RN 848251-94-5 HCA

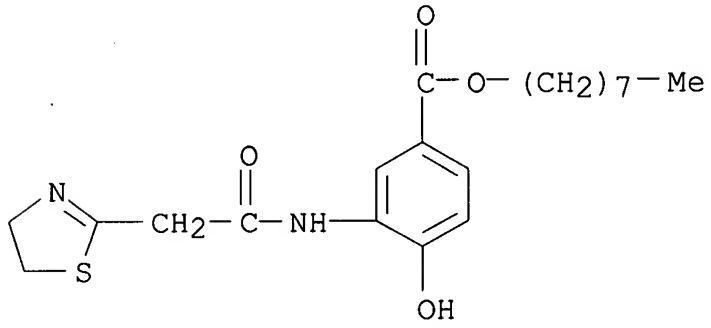
CN Benzoic acid, 3-[[(4,5-dihydro-2-thiazolyl)acetyl]amino]-4-hydroxy-, 3,7-dimethyloctyl ester (9CI) (CA INDEX NAME)



IT **848251-93-4 848251-95-6 848251-96-7**
(**coupler**; heat-developable **diazo** recording material contg. anilide deriv. **coupler**)

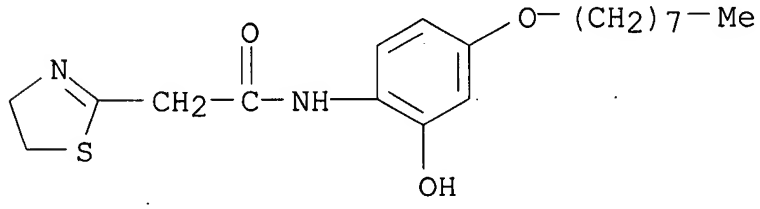
RN 848251-93-4 HCA

CN Benzoic acid, 3-[[(4,5-dihydro-2-thiazolyl)acetyl]amino]-4-hydroxy-, octyl ester (9CI) (CA INDEX NAME)

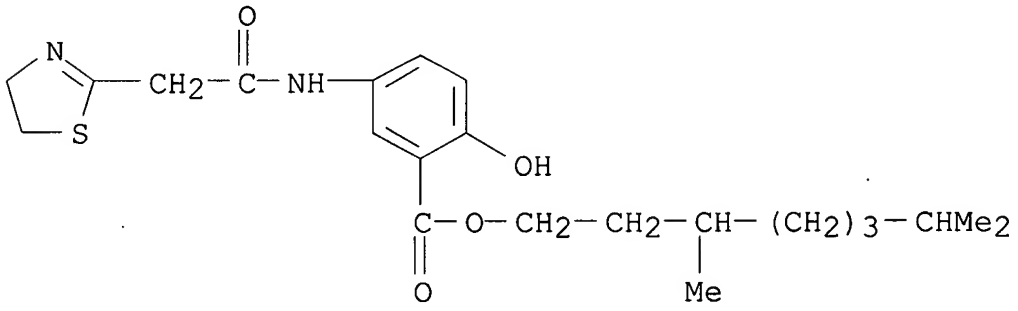


RN 848251-95-6 HCA

CN 2-Thiazoleacetamide, 4,5-dihydro-N-[2-hydroxy-4-(octyloxy)phenyl]- (9CI) (CA INDEX NAME)



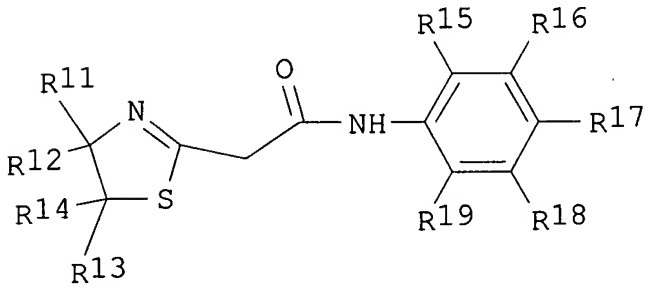
RN 848251-96-7 HCA
CN Benzoic acid, 5-[[(4,5-dihydro-2-thiazolyl)acetyl]amino]-2-hydroxy-, 3,7-dimethyloctyl ester (9CI) (CA INDEX NAME)



IC ICM B41M005-30
ICS B41M005-28
CC 74-10 (Radiation Chemistry, Photochemistry, and Photographic and Other Reprographic Processes)
ST heat developable **diazo** recording anilide **coupler**
IT **Diazo** process
(heat-developable; heat-developable **diazo** recording material contg. anilide deriv. **coupler**)
IT Polyureas
Polyurethanes, uses
(microcapsule shell; heat-developable **diazo** recording material contg. anilide deriv. **coupler**)
IT **848251-94-5P**
(**coupler**; heat-developable **diazo** recording material contg. anilide deriv. **coupler**)
IT **848251-93-4 848251-95-6 848251-96-7**
(**coupler**; heat-developable **diazo** recording material contg. anilide deriv. **coupler**)
IT 67928-21-6
(**diazo** compd.; heat-developable **diazo** recording material contg. anilide deriv. **coupler**)
IT 101-01-9
(heat-developable **diazo** recording material contg. anilide deriv. **coupler**)
IT 96-99-1, 4-Chloro-3-nitrobenzoic acid
(hydrolysis of; prepn. of anilide deriv. **coupler**)
IT 37337-02-3, Takenate D 110N
(microcapsule from; heat-developable **diazo** recording material contg. anilide deriv. **coupler**)
IT 616-82-0P, 4-Hydroxy-3-nitrobenzoic acid 848251-98-9P 848251-99-0P
(prepn. of anilide deriv. **coupler**)
IT 60-23-1, 2-Aminoethanethiol 106-21-8, Tetrahydrogeraniol

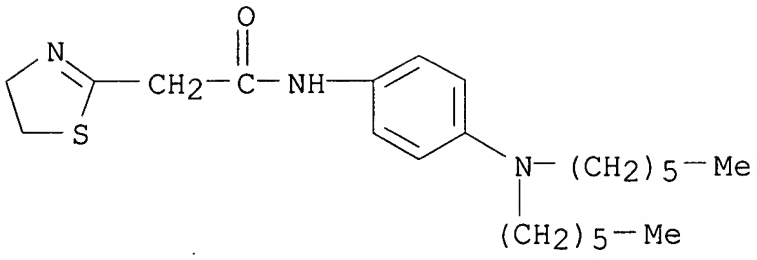
372-09-8, Cyanoacetic acid
(prepn. of anilide deriv. **coupler**)
IT 848251-97-8P
(redn. of; prepn. of anilide deriv. **coupler**)

L25 ANSWER 6 OF 16 HCA COPYRIGHT 2007 ACS on STN
142:186610 Heat-developable **diazo** recording material using specific **coupler**. Higuchi, Satoshi; Arioka, Daisuke; Ikeda, Takayoshi (Fuji Photo Film Co., Ltd., Japan). Jpn. Kokai Tokkyo Koho JP 2005028613 A 20050203, 42 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 2003-193339 20030708.
GI

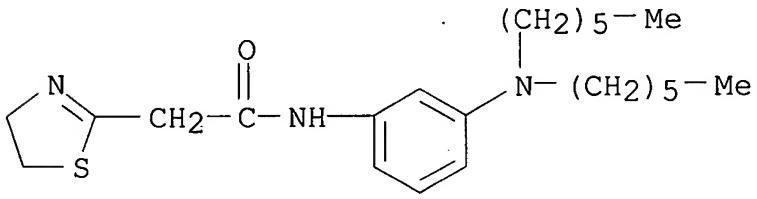


I

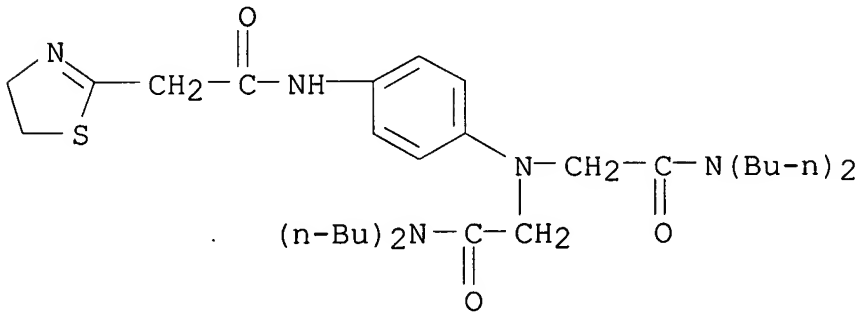
AB The material contains a **diazonium** compd. and a **coupler** I [R11-14 = H, alkyl, alkoxy, aryloxy, alkoxycarbonyl, aryloxycarbonyl, acyloxy, acyl, carbamoyl, acylamino, sulfamoyl, sulfonamide; R15-19 = H, halo, cyano, alkyl, aryl, alkoxy, aryloxy, alkylthio, arylthio, alkylsulfonyl, arylsulfonyl, alkoxycarbonyl, aryloxycarbonyl, acyloxy, acyl, carbamoyl, acylamino, sulfamoyl, sulfonamide, amino, hydrazino, hydroxyamino,urea, thiourea; ≥1 of R16-18 has Hammett's σp ≤-0.30] or its tautomer. The material shows good storage stability and gives clear yellow images with lightfastness.
IT **833487-25-5 833487-26-6 833487-27-7 833487-28-8 833487-29-9 833487-30-2 833487-31-3**
(**coupler**; heat-developable **diazo** recording material using specific **coupler**)
RN 833487-25-5 HCA
CN 2-Thiazoleacetamide, N-[4-(dihexylamino)phenyl]-4,5-dihydro- (9CI) (CA INDEX NAME)



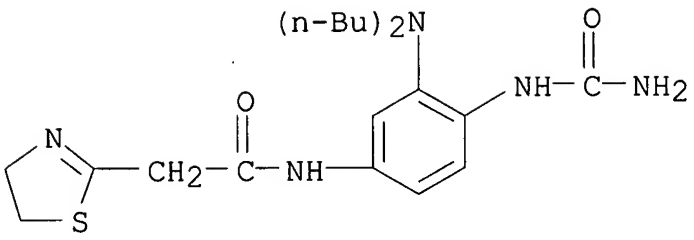
RN 833487-26-6 HCA
CN 2-Thiazoleacetamide, N-[3-(dihexylamino)phenyl]-4,5-dihydro- (9CI) (CA INDEX NAME)



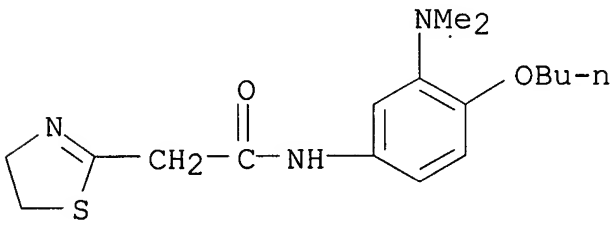
RN 833487-27-7 HCA
CN 2-Thiazoleacetamide, N-[4-[bis[2-(dibutylamino)-2-oxoethyl]amino]phenyl]-4,5-dihydro- (9CI) (CA INDEX NAME)



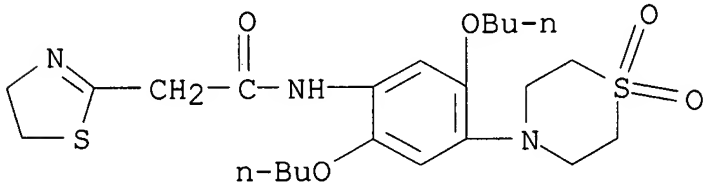
RN 833487-28-8 HCA
CN 2-Thiazoleacetamide, N-[4-[(aminocarbonyl)amino]-3-(dibutylamino)phenyl]-4,5-dihydro- (9CI) (CA INDEX NAME)



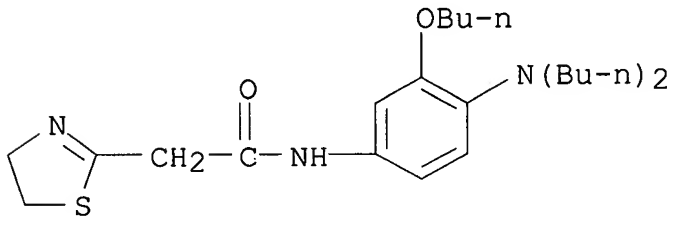
RN 833487-29-9 HCA
CN 2-Thiazoleacetamide, N-[4-butoxy-3-(dimethylamino)phenyl]-4,5-dihydro- (9CI) (CA INDEX NAME)



RN 833487-30-2 HCA
CN 2-Thiazoleacetamide, N-[2,5-dibutoxy-4-(1,1-dioxido-4-thiomorpholinyl)phenyl]-4,5-dihydro- (9CI) (CA INDEX NAME)



RN 833487-31-3 HCA
CN 2-Thiazoleacetamide, N-[3-butoxy-4-(dibutylamino)phenyl]-4,5-dihydro- (9CI) (CA INDEX NAME)



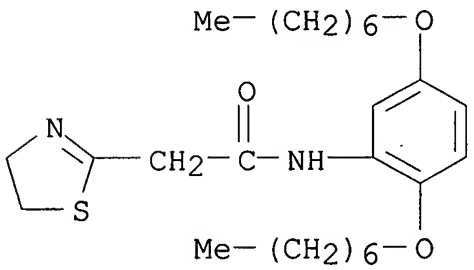
IC ICM B41M005-30
ICS B41M005-26; B41M005-28
CC 74-10 (Radiation Chemistry, Photochemistry, and Photographic and Other Reprographic Processes)
ST heat developable **diazo** recording material yellow **coupler**
IT **Diazo** process
(heat-developable; heat-developable **diazo** recording material using specific **coupler**)
IT Polyureas
Polyurethanes, uses
(microcapsule shell; heat-developable **diazo** recording material using specific **coupler**)
IT **833487-25-5 833487-26-6 833487-27-7 833487-28-8 833487-29-9 833487-30-2 833487-31-3**
(**coupler**; heat-developable **diazo** recording material using specific **coupler**)
IT 833487-33-5
(**diazonium** salt; heat-developable **diazo** recording material using specific **coupler**)
IT 101-01-9
(heat-developable **diazo** recording material using specific **coupler**)
IT 148130-89-6P
(microcapsule shell; heat-developable **diazo** recording material using specific **coupler**)

L25 ANSWER 7 OF 16 HCA COPYRIGHT 2007 ACS on STN
141:358159 Heat-developable **diazo** copying materials producing images with good lightfastness. Ikeda, Takayoshi; Saito, Naoki; Kanayama, Shuji (Fuji Photo Film Co., Ltd., Japan). Jpn. Kokai Tokkyo Koho JP 2004291477 A **20041021**, 52 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 2003-88531 20030327.
AB The materials have copying layers contg. azolinylacetic acids as **couplers** and **diazo** compds. sandwiched between ≥2 layers showing O permeability ≤20 mL/m2-day. Thus, a material comprising sequential layers of a PET film, an undercoating layer (O permeability 0.01 mL/m2-day), a cyan copying layer contg. **nondiazo** dye, an intermediate layer (O permeability 17 mL/m2-day), a magenta copying layer contg. a **diazonium** compd. and a **coupler**, an intermediate layer (O permeability 24 mL/m2-day), a yellow copying layer contg. a **diazonium** compd. and a **coupler**, a light transmission controlling layer (O permeability 4.3 mL/m2-day), and a protective layer (O permeability 152 mL/m2-day).
IT **769171-75-7**
(heat-developable **diazo** copying materials using

azolinylacetic acid **couplers** and producing images with good lightfastness)

RN 769171-75-7 HCA

CN 2-Thiazoleacetamide, N-[2,5-bis(heptyloxy)phenyl]-4,5-dihydro- (9CI) (CA INDEX NAME)



IC ICM B41M005-26
ICS B41M005-28; B41M005-30

CC 74-10 (Radiation Chemistry, Photochemistry, and Photographic and Other Reprographic Processes)

ST heat developable **diazo** azolinylacetic acid **coupler** ; lightfastness heat developable **diazo** copying

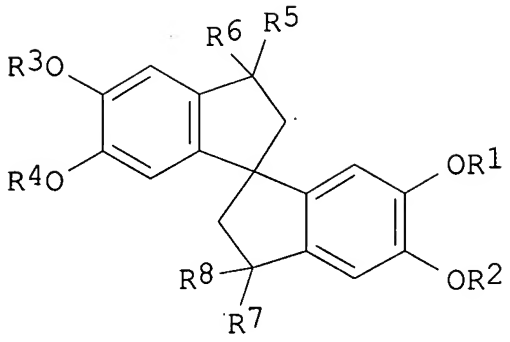
IT **Diazo** process
(heat-developable; heat-developable **diazo** copying materials using azolinylacetic acid **couplers** and producing images with good lightfastness)

IT 67928-21-6 186612-43-1 460350-61-2 663934-47-2 676461-94-2 **769171-75-7**
(heat-developable **diazo** copying materials using azolinylacetic acid **couplers** and producing images with good lightfastness)

L25 ANSWER 8 OF 16 HCA COPYRIGHT 2007 ACS on STN

141:322669 Heat-developable **diazo** recording materials. Ikeda, Takayoshi (Fuji Photo Film Co., Ltd., Japan). Jpn. Kokai Tokkyo Koho JP 2004276293 A **20041007**, 52 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 2003-67630 20030313.

GI



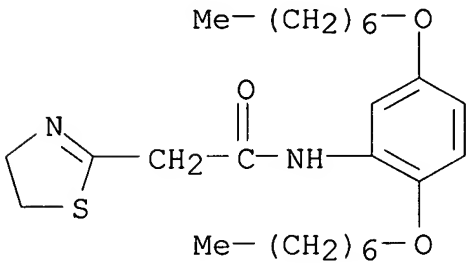
I

AB The material has a recording layer contg. an azolylacetic acid deriv., a **diazo** compd., and I (R1-4 = alkyl, aryl, heterocycle; R5-8 = H, alkyl, aryl; R5 and R6, and R7 and R8 may form a ring). It shows improved light stability of images.

IT **769171-75-7 769171-78-0**
(**coupler**; heat-developable **diazo** recording material contg. azolylacetic acid **coupler** and spiroindane compd.)

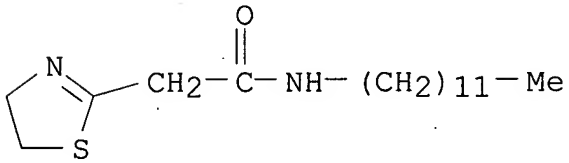
RN 769171-75-7 HCA

CN 2-Thiazoleacetamide, N-[2,5-bis(heptyloxy)phenyl]-4,5-dihydro- (9CI) (CA INDEX NAME)



RN 769171-78-0 HCA

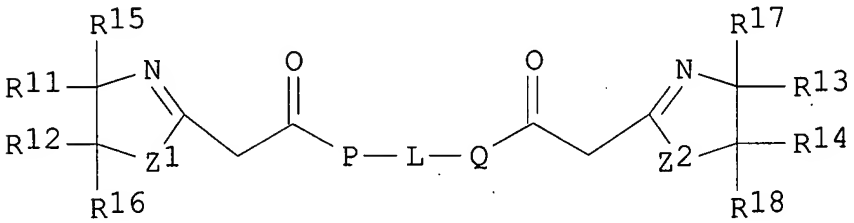
CN 2-Thiazoleacetamide, N-dodecyl-4,5-dihydro- (9CI) (CA INDEX NAME)



IC ICM B41M005-26
ICS B41M005-28; B41M005-30

CC 74-10 (Radiation Chemistry, Photochemistry, and Photographic and Other Reprographic Processes)
ST heat developable **diazo** recording material spiro indane compd; azolylacetic acid **coupler** **diazo** recording material
IT **Diazo** process
(heat-developable; heat-developable **diazo** recording material contg. azolylacetic acid **coupler** and spiroindane compd.)
IT **769171-75-7** **769171-78-0**
(**coupler**; heat-developable **diazo** recording material contg. azolylacetic acid **coupler** and spiroindane compd.)
IT 67928-21-6 186612-43-1 663934-47-2 676461-94-2
(**diazo** compd.; heat-developable **diazo** recording material contg. azolylacetic acid **coupler** and spiroindane compd.)
IT 89929-65-7
(heat-developable **diazo** recording material contg. azolylacetic acid **coupler** and spiroindane compd.)

L25 ANSWER 9 OF 16 HCA COPYRIGHT 2007 ACS on STN
141:233287 Novel azonitrile acetate derivative **coupler** and recording material containing the same with improved storage stability. Fujita, Akinori; Saito, Naoki; Takeuchi, Yosuke; Higuchi, Satoshi; Arioka, Daisuke; Ikeda, Takayoshi (Fuji Photo Film Co., Ltd., Japan). Jpn. Kokai Tokkyo Koho JP 2004244316 A **20040902**, 39 pp. (Japanese). CODEN: JKXXAF. APPLICATION: JP 2003-32491 20030210.
GI



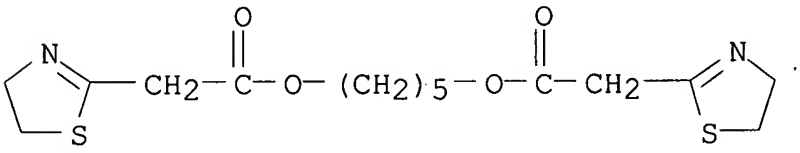
I

AB Disclosed is the novel azonitrile acetate deriv. **coupler** which is represented by I (R11-18 = H, alkyl, aryl, etc.; P, Q = single bond, O, amino; L = single bond, divalent org. group; and Z1,2 = S, O, etc.;). Also disclosed is the recording material

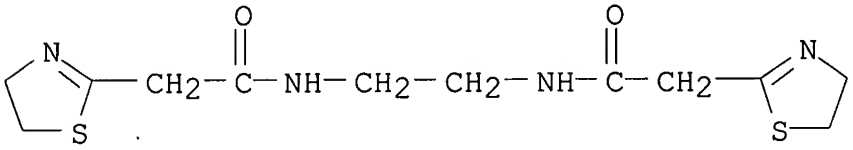
contg. said **coupler** and a **diazonium** salt, in which the **diazonium** salt is encapsulated in a microcapsule.

IT **748795-41-7P 748795-42-8P 748795-43-9P 748795-44-0P**
(novel azonitrile acetate deriv. **coupler** for recording material with improved storage stability)

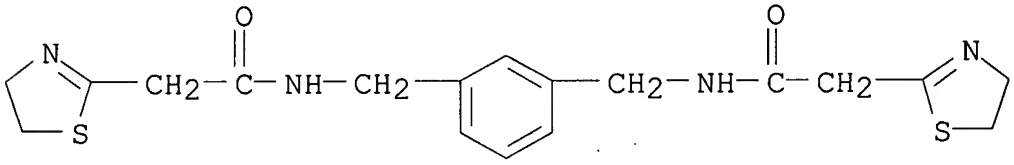
RN 748795-41-7 HCA
CN 2-Thiazoleacetic acid, 4,5-dihydro-, 1,5-pentanediyl ester (9CI) (CA INDEX NAME)



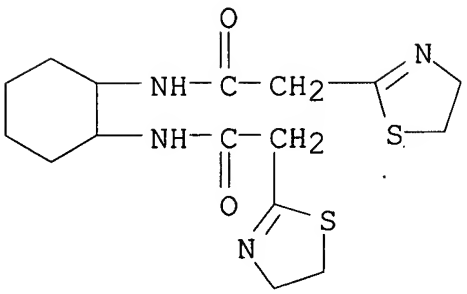
RN 748795-42-8 HCA
CN 2-Thiazoleacetamide, N,N'-1,2-ethanediylbis[4,5-dihydro- (9CI) (CA INDEX NAME)



RN 748795-43-9 HCA
CN 2-Thiazoleacetamide, N,N'-[1,3-phenylenebis(methylene)]bis[4,5-dihydro- (9CI) (CA INDEX NAME)



RN 748795-44-0 HCA
CN 2-Thiazoleacetamide, N,N'-1,2-cyclohexanediylbis[4,5-dihydro- (9CI) (CA INDEX NAME)



IC ICM C07D277-10
ICS B41M005-28; B41M005-30; C07D277-82; C07D207-20; C07D211-70; C07D233-26; C07D263-14; C07D263-56; C07D277-12

CC 74-10 (Radiation Chemistry, Photochemistry, and Photographic and Other Reprographic Processes)
Section cross-reference(s): 41

ST azonitrile acetate deriv **coupler diazonium** salt recording material

IT **Diazo** process
(novel azonitrile acetate deriv. **coupler** for recording material with improved storage stability)

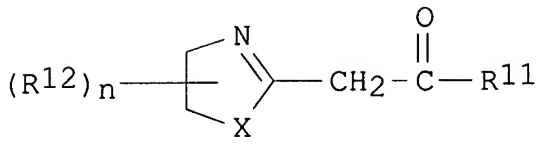
IT **748795-41-7P 748795-42-8P 748795-43-9P 748795-44-0P**
(novel azonitrile acetate deriv. **coupler** for recording material with improved storage stability)

IT 75-75-2, Methanesulfonic acid 105-56-6, Ethyl cyanoacetate 107-15-3, Ethylene diamine, reactions 372-09-8, Cyanoacetic acid 1477-55-0, 1,3-Benzenedimethanamine 29256-90-4, Diaminocyclohexane
(novel azonitrile acetate deriv. **coupler** for recording material with improved storage stability)

U.S.

L25 ANSWER 10 OF 16 HCA COPYRIGHT 2007 ACS on STN
141:197403 Azolinyl acetic acid derivative and azolinyl acetic acid derivative containing recording material. Saito, Naoki; Matsushita, Tetsunori; Fujita, Akinori; Takeuchi, Yohsuke; Higuchi, Satoshi; Ikeda, Kimi (Fuji Photo Film Co., Ltd., Japan). U.S. Pat. Appl. Publ. US 2004157157 A1 **20040812**, 25 pp. (English).
CODEN: USXXCO. APPLICATION: US 2004-773366 20040209. PRIORITY: JP 2003-32490 20030210.

GI



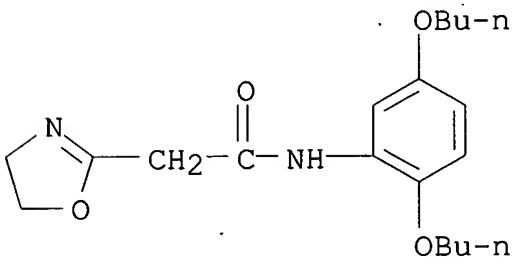
I

AB The present invention relates to a thermal recording material having, on a support, a recording layer contg. an azolinyl acetic acid deriv. and a diazo compd. The azolinyl acetic acid deriv. is preferably is a compd. represented by formula I (X = O, S; R11 = alkyl group, an aryl group, a heterocyclic group, -OR13 or -NR14R15; R12 = a substituent; R13 = an alkyl group, an aryl group or a heterocyclic group; R14,15 = H, an alkyl group, an aryl group or a heterocyclic group; n = 0-4; and, when n≥ 2, two or more R12s may be linked with each other to form a ring).

IT **737767-86-1P 737767-87-2P 737767-89-4P 737767-90-7P**
(azolinyl acetic acid deriv. for thermal recording material)

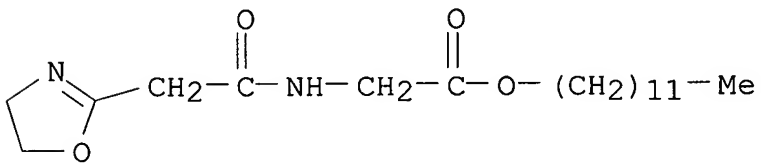
RN 737767-86-1 HCA

CN 2-Oxazoleacetamide, N-(2,5-dibutoxyphenyl)-4,5-dihydro- (9CI) (CA INDEX NAME)



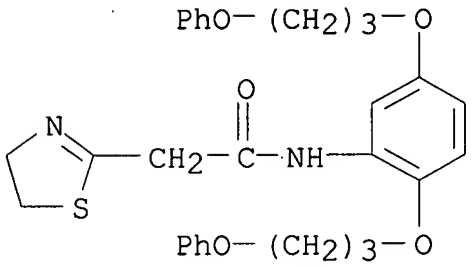
RN 737767-87-2 HCA

CN Glycine, N-[(4,5-dihydro-2-oxazolyl)acetyl]-, dodecyl ester (9CI) (CA INDEX NAME)

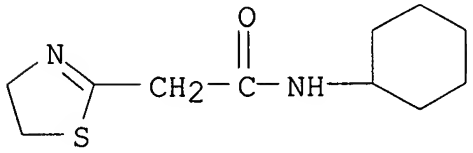


RN 737767-89-4 HCA

CN 2-Thiazoleacetamide, N-[2,5-bis(3-phenoxypropoxy)phenyl]-4,5-dihydro- (9CI) (CA INDEX NAME)



RN 737767-90-7 HCA
CN 2-Thiazoleacetamide, N-cyclohexyl-4,5-dihydro- (9CI) (CA INDEX NAME)



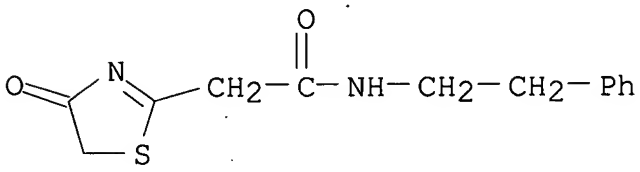
IC ICM G03C001-492
INCL 430270100
CC 74-7 (Radiation Chemistry, Photochemistry, and Photographic and Other Reprographic Processes)
IT **737767-86-1P 737767-87-2P** 737767-88-3P
737767-89-4P 737767-90-7P 737767-91-8P
(azolinyl acetic acid deriv. for thermal recording material)

L25 ANSWER 11 OF 16 HCA COPYRIGHT 2007 ACS on STN
138:254633 Mechanism of stereoselective synthesis of push-pull (Z)-4-oxothiazolidine derivatives containing an exocyclic double bond. A MNDO-PM3 study. Markovic, Rade; Vitnik, Zeljko; Baranac, Marija; Juranic, Ivan (Faculty of Chemistry, University of Belgrade, Belgrade, 11001, Yugoslavia). Journal of Chemical Research, Synopses (10), 485-489 (English) **2002**. CODEN: JRPSDC. ISSN: 0308-2342. OTHER SOURCES: CASREACT 138:254633. Publisher: Science Reviews.
AB Calcns. using the MNDO-PM3 method were performed to elucidate the mechanism of stereoselective base-catalyzed reaction affording exclusively (Z)-2-alkylidene-4-oxothiazolidine push-pull derivs. from the corresponding a-mercapto esters and activated β-oxonitriles in ethanol as a solvent.
IT **502764-97-8**
(mechanism of stereoselective synthesis of push-pull

(Z)-4-oxothiazolidine derivs. contg. exocyclic double bond and MNDO-PM3 study)

RN 502764-97-8 HCA

CN 2-Thiazoleacetamide, 4,5-dihydro-4-oxo-N-(2-phenylethyl)- (9CI) (CA INDEX NAME)



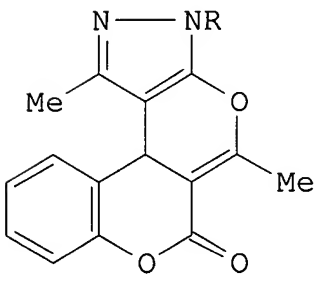
CC 22-2 (Physical Organic Chemistry)

IT 185739-19-9 502764-93-4 502764-94-5 502764-95-6 502764-96-7 **502764-97-8** 502764-98-9
(mechanism of stereoselective synthesis of push-pull (Z)-4-oxothiazolidine derivs. contg. exocyclic double bond and MNDO-PM3 study)

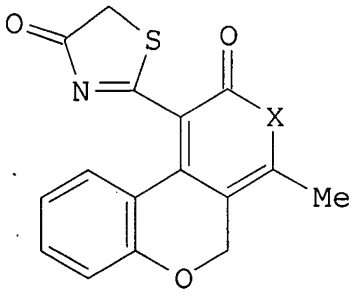
L25 ANSWER 12 OF 16 HCA COPYRIGHT 2007 ACS on STN

118:80855 Reactions with coumarin derivatives: synthesis of several new coumarinopyrazoles, coumarinopyridines and coumarinyl azoles. Ismail, Nabila A. (Fac. Sci., Zagazig Univ., Zagazig, Egypt). Egyptian Journal of Pharmaceutical Sciences, 32(3-4), 685-93 (English) **1991**. CODEN: EJPSBZ. ISSN: 0301-5068.

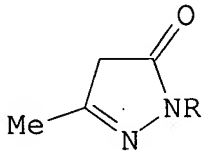
GI



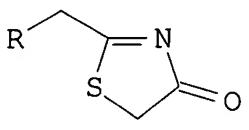
I



II



III



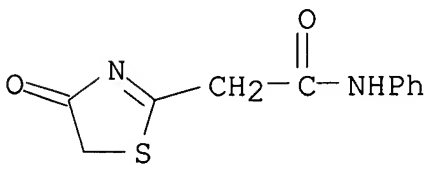
IV

AB New coumarinopyranopyrazoles I (R = H, Ph), and thiazolylcoumarinopyridine II (X = NPh) and -coumarinopyranone II (X = O) were synthesized via the reactions of 3-acetylcoumarin with different active methylene heterocyclic derivs. III (R = H, Ph) and IV (R = CONHPh, CO2Et) resp. Various other coumarin derivs. were also prepd. by the reactions of 3-(bromoacetyl)coumarin with III and IV (R = CN, CO2Et, CONHPh).

IT **87007-72-5**
(cyclocondensation of, with acetylcoumarin)

RN 87007-72-5 HCA

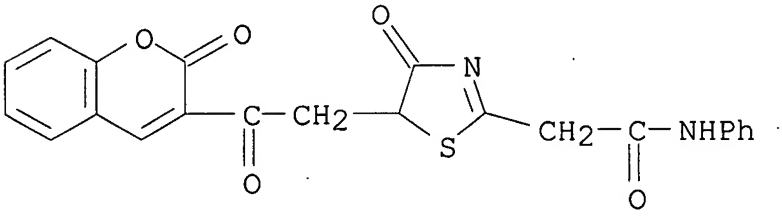
CN 2-Thiazoleacetamide, 4,5-dihydro-4-oxo-N-phenyl- (9CI) (CA INDEX NAME)



IT **145615-49-2P**
(prepn. of)

RN 145615-49-2 HCA

CN 2-Thiazoleacetamide, 4,5-dihydro-4-oxo-5-[2-oxo-2-(2-oxo-2H-1-benzopyran-3-yl)ethyl]-N-phenyl- (9CI) (CA INDEX NAME)



CC 28-8 (Heterocyclic Compounds (More Than One Hetero Atom))
Section cross-reference(s): 27

IT 89-25-8 108-26-9 877-87-2 **87007-72-5**
(cyclocondensation of, with acetylcoumarin)

IT 145615-41-4P 145615-42-5P 145615-43-6P 145615-44-7P
145615-45-8P 145615-46-9P 145615-47-0P 145615-48-1P
145615-49-2P 145639-99-2P
(prepn. of)

L25 ANSWER 13 OF 16 HCA COPYRIGHT 2007 ACS on STN

116:174042 Heterocycles synthesis through reactions of nucleophiles with acrylonitriles. Part V. Synthesis of several new thiazole and thiazolo[2,3-a]pyridine derivatives. Abdel-Latif, F. F.; Shaker, R.

M. (Fac. Sci., El-Minia Univ., El-Minia, Egypt). Polish Journal of Chemistry, 65(5-6), 1043-8 (English) **1991**. CODEN: PJCHDQ. ISSN: 0137-5083. OTHER SOURCES: CASREACT 116:174042.

GI

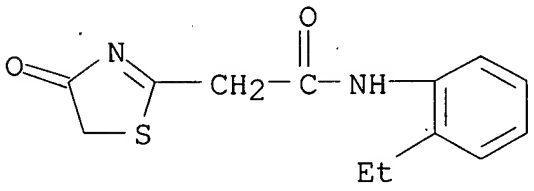
* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB RCH:CR1CN (I, R = 2-thienyl, 2-furyl, 1-naphthyl; R1 = cyano) reacted with [(ethylphenyl)carbamoyl]methylthiazolinone II in pyridine to give thiazolopyridines III. In contrast, 2-oxoindolin-3-ylidenemalononitrile and I [R = 2-thienyl, 2-furyl, R1 = COPh] reacted with II to give condensation products IV and V, resp. I (R = 2-thienyl, 2-furyl, R1 = CO2Et) cyclocondensed with II to give dioxotetrahydropyridylthiazolinones VI.

IT **129014-24-0P**
(prepn., condensation, cyclocondensation, and cycloaddn. reaction of, with acrylonitrile derivs.)

RN 129014-24-0 HCA

CN 2-Thiazoleacetamide, N-(2-ethylphenyl)-4,5-dihydro-4-oxo- (9CI) (CA INDEX NAME)



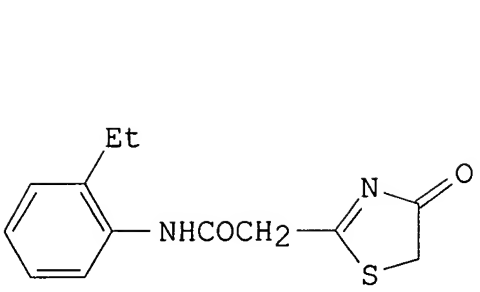
CC 28-7 (Heterocyclic Compounds (More Than One Hetero Atom))

IT **129014-24-0P**
(prepn., condensation, cyclocondensation, and cycloaddn. reaction of, with acrylonitrile derivs.)

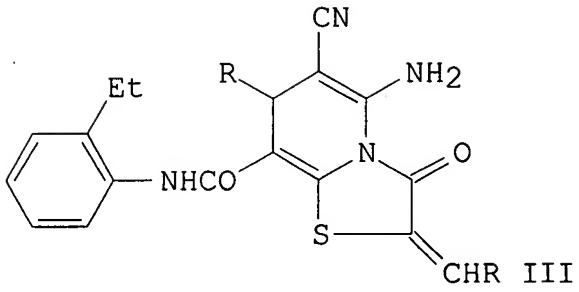
L25 ANSWER 14 OF 16 HCA COPYRIGHT 2007 ACS on STN

113:115161 Heterocycle synthesis through reactions of nucleophiles with acrylonitriles. Part 5. Synthesis of several new thiazole and thiazolo[2,3-a]pyridine derivatives. Abdel-Latif, F. F.; Shaker, R. M. (Fac. Sci., El-Minia Univ., El-Minia, Egypt). Phosphorus, Sulfur and Silicon and the Related Elements, 48(1-4), 217-21 (English) **1990**. CODEN: PSSLEC. ISSN: 1042-6507. OTHER SOURCES: CASREACT 113:115161.

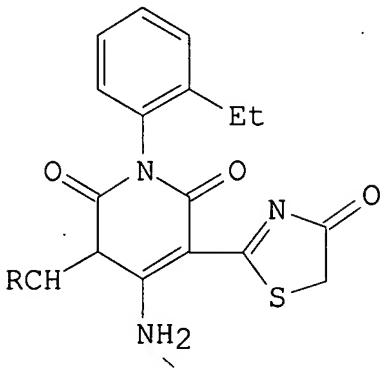
GI



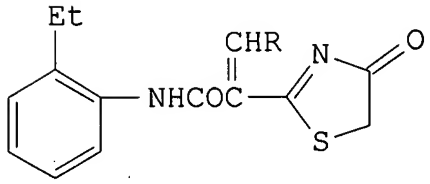
II



III



IV



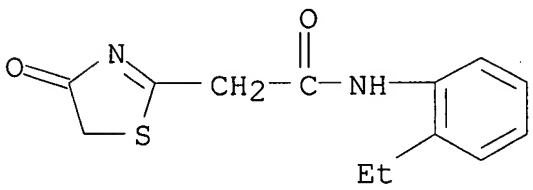
V

AB Reaction of RCH:CR1CN (I; R = 2-thienyl, 2-furyl, 1-naphthyl; R1 = CN) with thiazolinonylacetamide II gave thiazolopyridines III. Similar reactions with I (R = 2-thienyl, 2-furyl; R1 = CO2Et) gave thiazolinonylpyridinones IV, while with I (R = 2-indolonyl, R1 = CN; R = 2-thienyl, 2-furyl; R1 = COPh) ylidene derivs., e.g. V, were obtained.

IT **129014-24-0P**
(prepn. and reactions of, with acrylonitriles, thiazolopyridines, pyridinones, and ylidene derivs. from)

RN 129014-24-0 HCA

CN 2-Thiazoleacetamide, N-(2-ethylphenyl)-4,5-dihydro-4-oxo- (9CI) (CA INDEX NAME)



CC 28-7 (Heterocyclic Compounds (More Than One Hetero Atom))

IT **129014-24-0P**

(prepn. and reactions of, with acrylonitriles, thiazolopyridines, pyridinones, and ylidene derivs. from)

L25 ANSWER 15 OF 16 HCA COPYRIGHT 2007 ACS on STN
110:57610 Activated nitriles in heterocyclic synthesis: a novel synthesis of fused pyrimidine, pyrazole and thiazole derivatives. Ibrahim, Mohamed Kamal Ahmed (Fac. Sci., Cairo Univ., Giza, Egypt). Indian Journal of Chemistry, Section B: Organic Chemistry Including Medicinal Chemistry, 27B(5), 478-81 (English) **1988**. CODEN: IJSBDB. ISSN: 0376-4699. OTHER SOURCES: CASREACT 110:57610.
GI

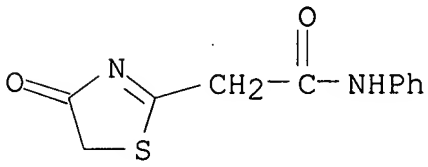
* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB Treating p-MeOC6H4CH:C(CN)2 (I) with pyrazolone II (R = H, R1 = NH2) gave pyrazolo[1,5-a]pyrimidine III, whereas the treating I with II (R = Cl, R1 = OH) gave pyrazolo[1,5-b][1,3]oxazine IV. Pyrimido[1,2-a]benzimidazole V was prepd. by treating I with 2-aminobenzimidazole. Treating I with 3-aminotriazole gave s-triazolo[3,4-b]pyrimidine VI. 7H-Thiazolo[3,2-a]pyridine VII was prepd. by treating I with thiazolinone VIII.

IT **87007-72-5**
(reaction of, with anisylidenemalononitrile)

RN 87007-72-5 HCA

CN 2-Thiazoleacetamide, 4,5-dihydro-4-oxo-N-phenyl- (9CI) (CA INDEX NAME)



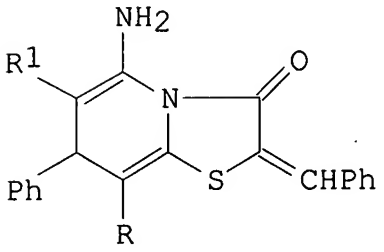
CC 28-16 (Heterocyclic Compounds (More Than One Hetero Atom))

IT 61-82-5, 1H-1,2,4-Triazol-3-amine 62-56-6, Thiourea, reactions 100-19-6 934-32-7, 2-Aminobenzimidazole 3656-02-8 3656-10-8 **87007-72-5** 93032-54-3 118464-16-7
(reaction of, with anisylidenemalononitrile)

L25 ANSWER 16 OF 16 HCA COPYRIGHT 2007 ACS on STN
99:105167 Activated nitriles in heterocyclic synthesis: the reaction of nitriles with mercapto acids. Elgemeie, Galal Eldin Hamza; Elfahham, Hassan Attia; Hassan, Sanna Mohy Eldin; Elnagdi, Mohamed Hilmy (Fac. Sci., Minia Univ., Minia, Egypt). Zeitschrift fuer

Naturforschung, Teil B: Anorganische Chemie, Organische Chemie, 38B(6), 781-5 (English) **1983**. CODEN: ZNBAD2. ISSN: 0340-5087. OTHER SOURCES: CASREACT 99:105167.

GI



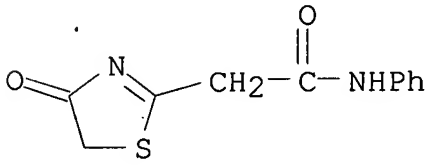
I

AB New thiazolo[2,3-a]pyridines I (R = cyano, CO2Et, CONHPh, R1 = cyano; R = CONHPh, R1 = CO2Et) were obtained via the reaction of HSC(:CHPh)CO2H and HSCH2CO2H with some activated nitriles and treatment of the resulting 2-thiazolin-4-ones with PhCH:CR1CN.

IT **87007-72-5P**
(prepn. and reaction of, with benzylidenemalononitrile)

RN 87007-72-5 HCA

CN 2-Thiazoleacetamide, 4,5-dihydro-4-oxo-N-phenyl- (9CI) (CA INDEX NAME)



CC 28-7 (Heterocyclic Compounds (More Than One Hetero Atom))

IT **87007-72-5P**
(prepn. and reaction of, with benzylidenemalononitrile)

=> D L26 1-13 CBIB ABS HITSTR HITIND

L26 ANSWER 1 OF 13 HCA COPYRIGHT 2007 ACS on STN

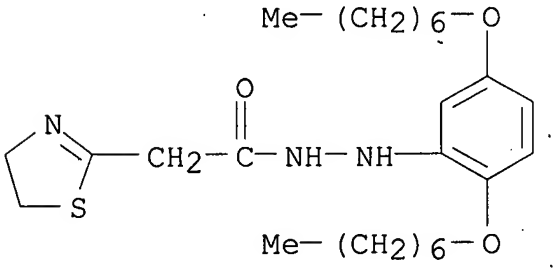
143:68419 **Diazo recording** material containing acylhydrazide **coupler** and **diazo** compound.
Saito, Naoki; Ikeda, Takayoshi (Fuji Photo Film Co., Ltd., Japan).
Jpn. Kokai Tokkyo Koho JP 2005161698 A 20050623, 38 pp.
(Japanese). CODEN: JKXXAF. APPLICATION: JP 2003-404259 20031203.

AB The material has a **recording** layer contg. the acylhydrazide **coupler** and the **diazo** compd. on a support. The material shows improved color developability, raw stock stability, and light stability and reduced background stain.

IT **854089-11-5 854089-13-7 854089-15-9 854089-17-1 854089-25-1 854089-27-3**
(**coupler; diazo recording** material contg. acylhydrazide **coupler** and **diazo** compd.)

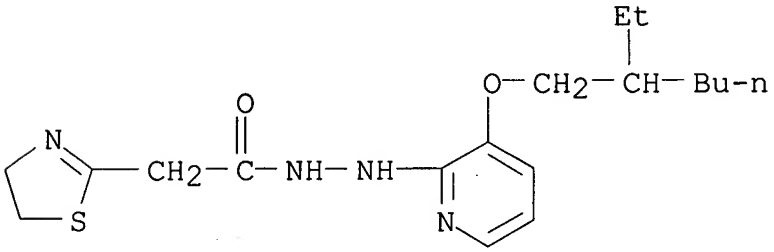
RN 854089-11-5 HCA

CN 2-Thiazoleacetic acid, 4,5-dihydro-, 2-[2,5-bis(heptyloxy)phenyl]hydrazide (9CI) (CA INDEX NAME)



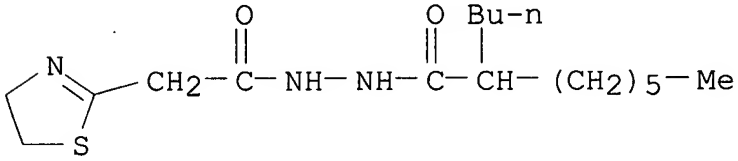
RN 854089-13-7 HCA

CN 2-Thiazoleacetic acid, 4,5-dihydro-, 2-[3-[(2-ethylhexyl)oxy]-2-pyridinyl]hydrazide (9CI) (CA INDEX NAME)

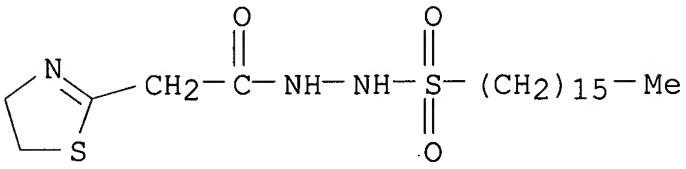


RN 854089-15-9 HCA

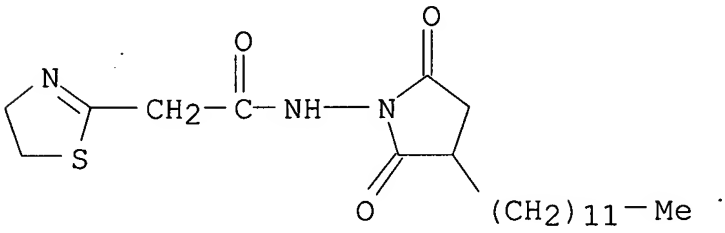
CN 2-Thiazoleacetic acid, 4,5-dihydro-, 2-(2-butyl-1-oxooctyl)hydrazide (9CI) (CA INDEX NAME)



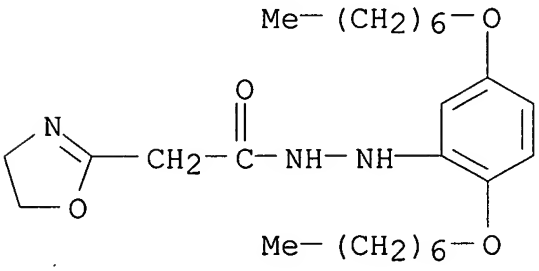
RN 854089-17-1 HCA
CN 2-Thiazoleacetic acid, 4,5-dihydro-, 2-(hexadecylsulfonyl)hydrazide (9CI) (CA INDEX NAME)



RN 854089-25-1 HCA
CN 2-Thiazoleacetamide, N-(3-dodecyl-2,5-dioxo-1-pyrrolidinyl)-4,5-dihydro- (9CI) (CA INDEX NAME)



RN 854089-27-3 HCA
CN 2-Oxazoleacetic acid, 4,5-dihydro-, 2-[2,5-bis(heptyloxy)phenyl]hydrazide (9CI) (CA INDEX NAME)



IC ICM B41M005-30
ICS B41M005-28
CC 74-10 (Radiation Chemistry, Photochemistry, and Photographic and Other Reprographic Processes)
ST **diazo recording** material acyl hydrazide **diazo** compd; heat developable **diazo recording** material
IT **Diazo** process
(heat-developable; **diazo recording** material contg. acylhydrazide **coupler** and **diazo**

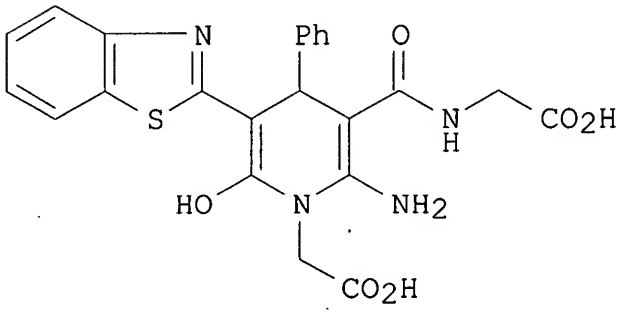
compd.)

IT **854089-11-5 854089-13-7 854089-15-9**
854089-17-1 854089-19-3 854089-21-7 854089-23-9
854089-25-1 854089-27-3 854089-29-5
(**coupler; diazo recording** material contg. acylhydrazide **coupler** and **diazo** compd.)

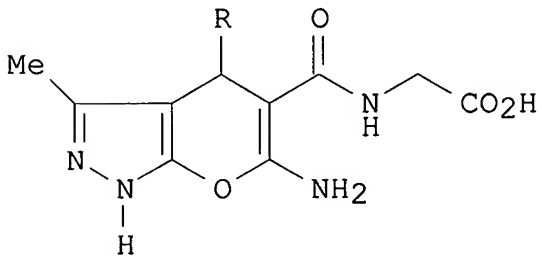
IT 854089-32-0
(**diazonium** salt; **diazo recording** material contg. acylhydrazide **coupler** and **diazo** compd.)

L26 ANSWER 2 OF 13 HCA COPYRIGHT 2007 ACS on STN
132:322108 Amino acid derivatives in organic synthesis, part 4: Facile synthesis of heterocycles containing a glycine residue. Chabaka, Laila M.; Allam, Yehia A.; Nawwar, Galal A. M. (Pesticides Laboratory, National Research Center, Cairo, Egypt). Zeitschrift fuer Naturforschung, B: Chemical Sciences, 55(1), 104-108 (English) **2000**. CODEN: ZNBSEN. ISSN: 0932-0776. OTHER SOURCES: CASREACT 132:322108. Publisher: Verlag der Zeitschrift fuer Naturforschung.

GI



I



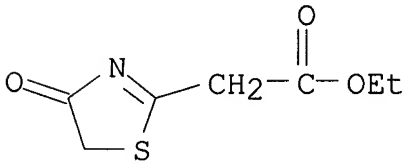
II

AB Pyridines, thiazolopyridines and pyrazolopyrans contg. glycinate residue, e.g. I and II (R = Ph, 4-chlorophenyl), were prepd. by reacting N-cyanoacryloylglycinate ylidenes with active methylene compds. via a Michael addn. - intracyclization synthetic pathway. Simple routes for the synthesis of heterocycles with an amino acid residue have been previously reported as the incorporation of these residues improves the pharmacokinetics and toxicity of active compds. However, trials to deesterify these residues for **coupling** purposes were unsuccessful. So, we tried herein new approaches for synthesizing heterocycles carrying one or two glycine moieties with free carboxylic acid group to facilitate further peptide linkage on one hand and on the other one could be able to form metal chelates, a property having a significant output on the toxicol. behavior.

IT **877-87-2 29182-42-1**
(prepn. of heterocycles contg. a glycine residue)

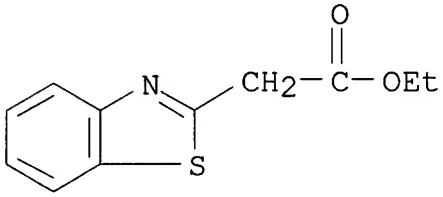
RN 877-87-2 HCA

CN 2-Thiazoleacetic acid, 4,5-dihydro-4-oxo-, ethyl ester (9CI) (CA INDEX NAME)



RN 29182-42-1 HCA

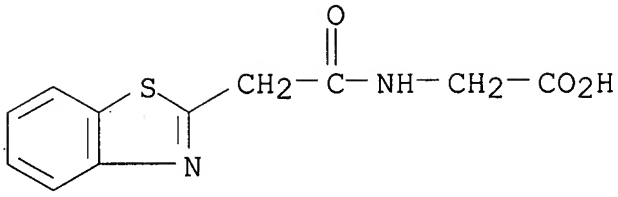
CN 2-Benzothiazoleacetic acid, ethyl ester (8CI, 9CI) (CA INDEX NAME)



IT **267240-10-8P**
(prepn. of heterocycles contg. a glycine residue)

RN 267240-10-8 HCA

CN Glycine, N-(2-benzothiazolylacetyl)- (9CI) (CA INDEX NAME)



CC 34-2 (Amino Acids, Peptides, and Proteins)
Section cross-reference(s): 28

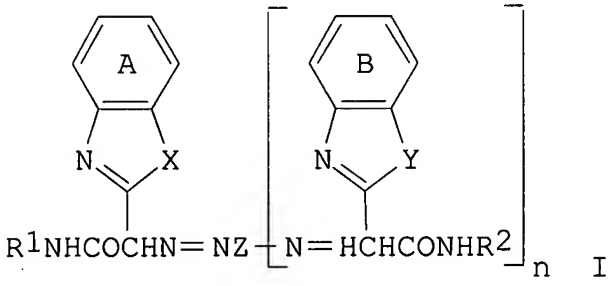
IT 98-01-1, 2-Furaldehyde, reactions 98-03-3, Thiophene-2-aldehyde 100-34-5, Phenyl **diazonium** chloride 105-56-6 108-26-9 109-77-3, Malononitrile **877-87-2** 15743-44-9, Potassium glycinate **29182-42-1** 267240-09-5
(prepn. of heterocycles contg. a glycine residue)

IT 117580-69-5P 267240-00-6P 267240-07-3P **267240-10-8P** 267240-11-9P 267240-14-2P
(prepn. of heterocycles contg. a glycine residue)

L26 ANSWER 3 OF 13 HCA COPYRIGHT 2007 ACS on STN

116:43076 Azo pigments, their preparation and use. Jung, Ruediger; Deubel, Reinhold (Hoechst A.-G., Germany). Ger. Offen. DE 4007535 A1 **19910912**, 45 pp. (German). CODEN: GWXXBX.
APPLICATION: DE 1990-4007535 19900309.

GI



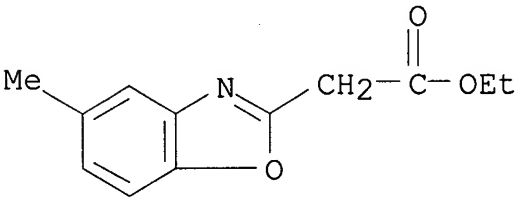
AB The pigments (I; R1, R2 = carbocyclic or heterocyclic arom. group; X, Y = O, NH, NR3; R3 = aliph. or arom. group; n = 0, 1; Z = arom. **diazo** or **bisdiazo** component residue; rings A and B may be substituted or annelated) are prepd. by azo **coupling** and are suitable for plastics, textiles, and paper. Thus, Et 2-benzimidazolylacetate was condensed with 2-aminoanisole to give the methoxyanilide (II). 4-Chloro-2-nitroaniline was

diazotized and **coupled** with II to give reddish yellow I (R1 = o-methoxyphenyl; X = NH; Z = 4-chloro-2-nitrophenyl; n = 0).

IT **138399-44-7P 138420-09-4P**
(prepn. and amidation of)

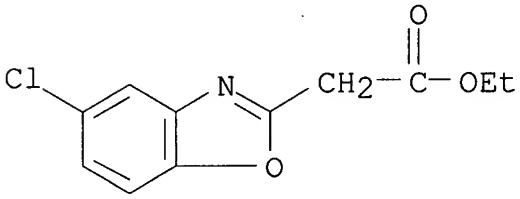
RN 138399-44-7 HCA

CN 2-Benzoxazoleacetic acid, 5-methyl-, ethyl ester (9CI) (CA INDEX NAME)



RN 138420-09-4 HCA

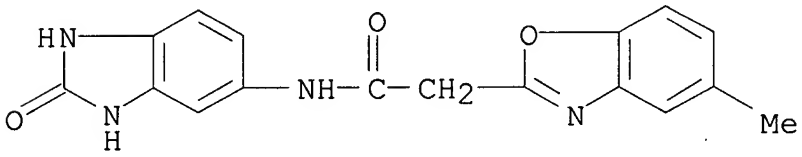
CN 2-Benzoxazoleacetic acid, 5-chloro-, ethyl ester (9CI) (CA INDEX NAME)



IT **138399-32-3P 138399-39-0P 138399-40-3P**
138399-41-4P 138399-42-5P
(prepn. of, as azo **coupling** component)

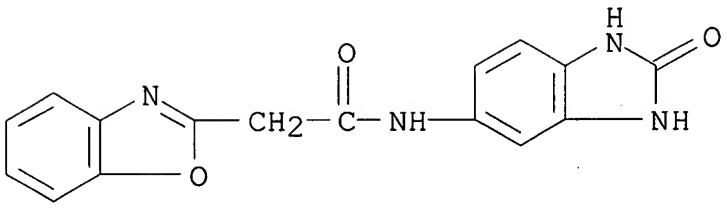
RN 138399-32-3 HCA

CN 2-Benzoxazoleacetamide, N-(2,3-dihydro-2-oxo-1H-benzimidazol-5-yl)-5-methyl- (9CI) (CA INDEX NAME)

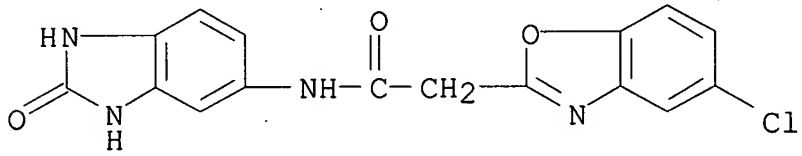


RN 138399-39-0 HCA

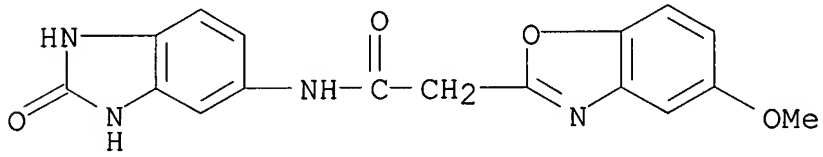
CN 2-Benzoxazoleacetamide, N-(2,3-dihydro-2-oxo-1H-benzimidazol-5-yl)- (9CI) (CA INDEX NAME)



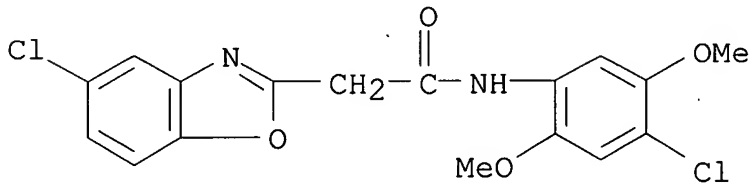
RN 138399-40-3 HCA
CN 2-Benzoxazoleacetamide, 5-chloro-N-(2,3-dihydro-2-oxo-1H-benzimidazol-5-yl)- (9CI) (CA INDEX NAME)



RN 138399-41-4 HCA
CN 2-Benzoxazoleacetamide, N-(2,3-dihydro-2-oxo-1H-benzimidazol-5-yl)-5-methoxy- (9CI) (CA INDEX NAME)



RN 138399-42-5 HCA
CN 2-Benzoxazoleacetamide, 5-chloro-N-(4-chloro-2,5-dimethoxyphenyl)- (9CI) (CA INDEX NAME)



IC ICM C09B029-32
ICS C09B035-035; C09B056-12; C09B041-00; C09B063-00; D06P001-44; C09D017-00; C08K005-34; D01F001-04; B41M001-00
ICA C09D011-02; C09B067-10; C09B067-20; C09B067-22; D06P003-60; D06P003-40; D06P003-52; D06P003-24; C08K005-3447; C08K005-353

CC 41-3 (Dyes, Organic Pigments, Fluorescent Brighteners, and Photographic Sensitizers)

IT 88-17-5, 2-Aminobenzotrifluoride 88-53-9, 2-Amino-5-chloro-4-methylbenzenesulfonic acid 89-62-3, 4-Methyl-2-nitroaniline 89-63-4 95-23-8 95-82-9, 2,5-Dichloroaniline 96-96-8, 4-Methoxy-2-nitroaniline 97-52-9, 2-Methoxy-4-nitroaniline 99-27-4, Dimethyl 5-aminoisophthalate 99-52-5 99-55-8, 2-Methyl-5-nitroaniline 99-59-2, 2-Methoxy-5-nitroaniline 120-35-4, 3-Amino-4-methoxybenzanilide 134-20-3, Methyl 2-aminobenzoate 5372-81-6, Dimethyl aminoterephthalate 19694-10-1, 3-Amino-4-chlorobenzamide 49701-19-1 52298-44-9 67014-36-2 138399-43-6
(**coupling** of **diazotized**, with heterocyclic compds.)

IT 67499-07-4, 1,2-Bis(2-amino-5-nitrophenoxy)ethane 67499-48-3, 1,2-Bis(4-amino-3-nitrophenoxy)ethane
(**coupling** of tetrazotized, with heterocyclic compd.)

IT 91-94-1, 3,3'-Dichlorobenzidine 52411-34-4, 1,2-Bis(2-aminophenoxy)ethane
(**coupling** of tetrazotized, with heterocyclic compds.)

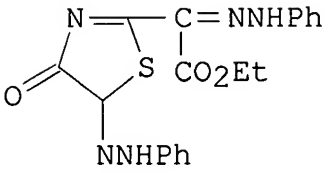
IT 52335-18-9P **138399-44-7P 138420-09-4P**
138420-10-7P
(prepn. and amidation of)

IT 138399-30-1P 138399-31-2P **138399-32-3P** 138399-33-4P
138399-34-5P 138399-35-6P 138399-36-7P 138399-37-8P
138399-38-9P **138399-39-0P 138399-40-3P**
138399-41-4P 138399-42-5P 138420-07-2P
138420-08-3P
(prepn. of, as azo **coupling** component)

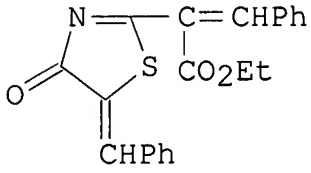
L26 ANSWER 4 OF 13 HCA COPYRIGHT 2007 ACS on STN

112:138951 Reaction of nitriles with mercaptoacetic acid. Facile synthesis of thiazolo[3,2-a]dihydropyridine and thiazolo[4,5-b]pyran derivatives. Ibrahim, Mohamed Kamal Ahmed (Faculty of Science, Cairo Univ., Giza, Egypt). Journal of the Indian Chemical Society, 66(6), 395-7 (English) **1989**. CODEN: JICSAH. ISSN: 0019-4522. OTHER SOURCES: CASREACT 112:138951.

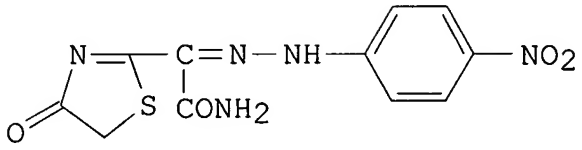
GI



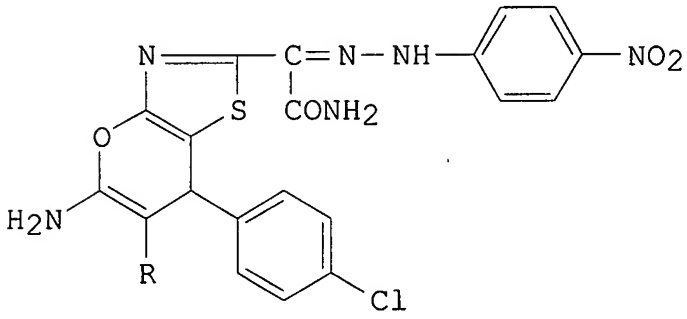
I



II



III



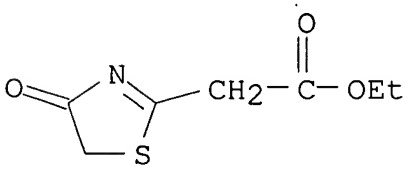
IV

AB 2-Ethoxycarbonylmethyl-4-thiazolinone reacted with 2 equiv each of **benzenediazonium** chloride and benzaldehyde to afford 4-thiazolinone derivs. (I and II, resp.). Treatment of II with malononitrile, cyanoacetamide, Et cyanoacetate, benzoylacetonitrile and cyanoacetanilide gave thiazolo[3,2-a]dihydropyridine derivs. Azocyanoacetamide reacted with mercaptoacetic acid in pyridine to yield 4-thiazolinone III, which was condensed with Et 4-chlorobenzylidenecyanoacetate and 4-chlorobenzylidenemalonitrile to furnish thiazolo[4,5-b]pyran derivs. IV (R = CO2Et, CN).

IT **877-87-2P**
(prepn. and **coupling** of, with **benzenediazonium** chlorides and benzaldehyde)

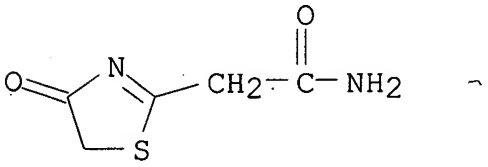
RN 877-87-2 HCA

CN 2-Thiazoleacetic acid, 4,5-dihydro-4-oxo-, ethyl ester (9CI) (CA INDEX NAME)



CC 28-7 (Heterocyclic Compounds (More Than One Hetero Atom))
IT **877-87-2P**
(prepn. and **coupling** of, with **benzenediazonium** chlorides and benzaldehyde)

L26 ANSWER 5 OF 13 HCA COPYRIGHT 2007 ACS on STN
110:116632 Synthesis of azo disperse dyes from activated nitriles for dyeing acetate and other fibers. Fadda, A. A.; Elmorsy, S. S.; Elagizy, S. A. (Fac. Sci., Mansoura Univ., Mansoura, Egypt). Indian Journal of Textile Research, 13(2), 87-91 (English) **1988**. CODEN: IJTRDU. ISSN: 0377-8436. OTHER SOURCES: CASREACT 110:116632.
AB 5,α-Bis(arylazo)-4-oxo-4,5-dihydro-1,3-thiazol-2-ylacetamides, α-(arylazo)cyanoacetic acid hydrazides, and 3-amino-4-(arylazo)-2-pyrazolin-5-ones (aryl = C6H4R, where R = H, Cl, NO2, Me, OH, OMe, CO2H, Br, Cl) were prepd. Azo-hydrazone tautomerism and the effect of the nature and position of the R groups in the **diazonium** components on the color were discussed.
IT **87947-93-1**
(**coupling** of, with **diazotized** aniline derivs.)
RN 87947-93-1 HCA
CN 2-Thiazoleacetamide, 4,5-dihydro-4-oxo- (9CI) (CA INDEX NAME)

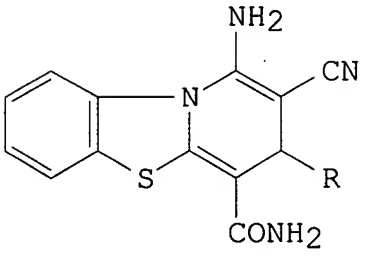


CC 41-3 (Dyes, Organic Pigments, Fluorescent Brighteners, and Photographic Sensitizers)
IT 96-96-8
(**coupling** of **diazotized**, with cyanoacethydrazide)
IT 108-44-1, reactions
(**coupling** of **diazotized**, with cyanoacethydrazide)
IT 118-92-3 536-90-3 554-00-7
(**coupling** of **diazotized**, with cyanoacethydrazide or oxodihydrothiazolacetamide)
IT 62-53-3, Benzenamine, reactions 88-74-4 95-55-6 100-01-6, reactions 104-94-9 106-40-1 106-47-8, reactions 106-49-0, reactions 108-42-9
(**coupling** of **diazotized**, with cyanoacethydrazide or oxodihydrothiazolacetamide)

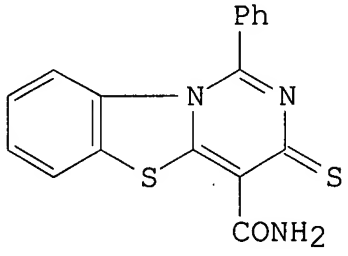
IT 140-87-4 **87947-93-1**
(**coupling** of, with **diazotized** aniline derivs.)

L26 ANSWER 6 OF 13 HCA COPYRIGHT 2007 ACS on STN
109:149468 Studies in sulfur heterocycles: novel synthesis of pyrido[2,1-b]benzothiazoles and pyrimido[6,1-b]benzothiazoles. Fathy, Nahed M.; Elgemeie, Galal E. H. (Appl. Org. Chem. Lab., Natl. Res. Cent., Dokki/Cairo, Egypt). Sulfur Letters, 7(5), 189-96 (English) **1988**. CODEN: SULED2. ISSN: 0278-6117. OTHER SOURCES: CASREACT 109:149468.

GI



I



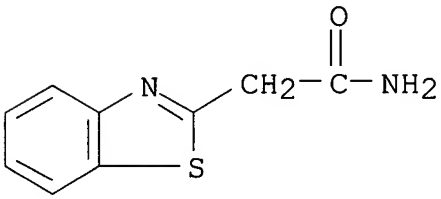
II

AB A novel synthesis of pyrido[2,1-b]benzothiazoles, e.g., I (R = 4-R1C6H4; R1 = H, Cl, Me, OMe), and pyrimido[6,1-b]benzothiazoles, e.g., II, from 2-benzothiazoleacetamide (III) is reported. Thus, condensation of III with RCHO followed by cyclocondensation with H2C(CN)2 gave I. Direct cyclocondensation of III with RCH:C(CN)2 gave 56-87% I.

IT **51542-41-7P**, 2-Benzothiazoleacetamide
(prepn. and condensation and cyclocondensation reactions of)

RN 51542-41-7 HCA

CN 2-Benzothiazoleacetamide (9CI) (CA INDEX NAME)



CC 28-16 (Heterocyclic Compounds (More Than One Hetero Atom))

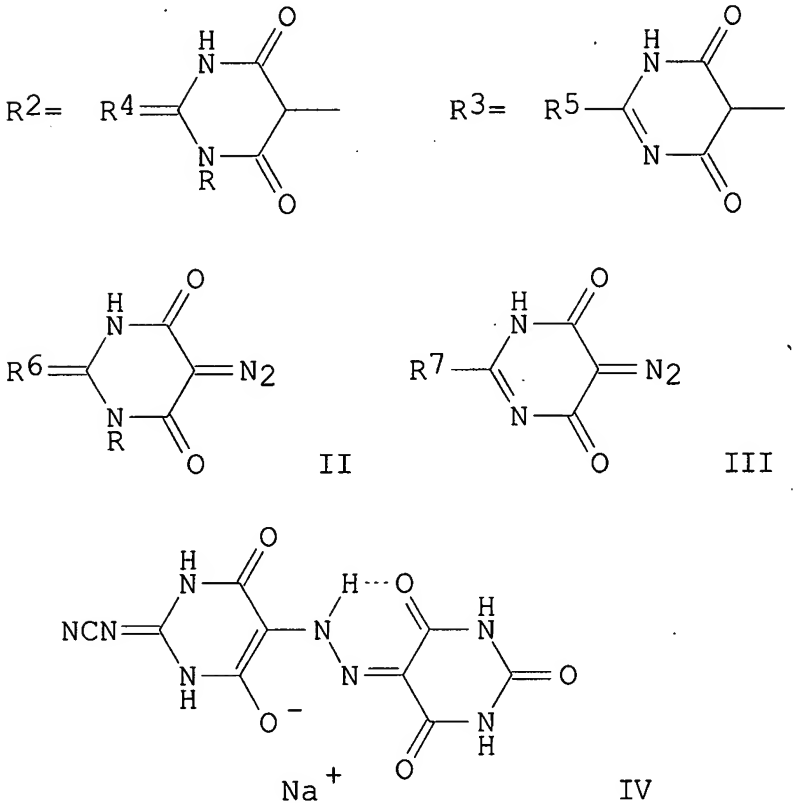
IT **51542-41-7P**, 2-Benzothiazoleacetamide
(prepn. and condensation and cyclocondensation reactions of)

IT 62-53-3, Aniline, reactions 104-94-9, 4-Methoxyaniline 106-47-8,

4-Chloroaniline, reactions 106-49-0, 4-Methylaniline, reactions
(sequential **diazotization** and **coupling**
reaction of, with benzothiazoleacetamide)

L26 ANSWER 7 OF 13 HCA COPYRIGHT 2007 ACS on STN
100:69857 Pyrimidine azo pigments. Lorenz, Manfred; Schuendehuette, Karl Heinz; Bornatsch, Wolfgang (Bayer A.-G., Fed. Rep. Ger.). Ger. Offen. DE 3307508 A1 **19831103**, 48 pp. (German). CODEN: GWXXBX. APPLICATION: DE 1983-3307508 19830303. PRIORITY: DE 1982-3215877 19820429.

GI



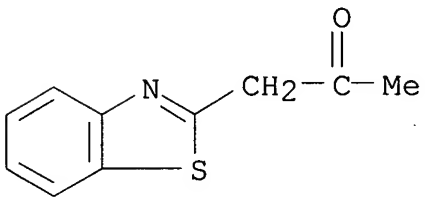
AB The title azo compds. RN:NR1 (I; R ≠ R1, R = R2 or R3 or their tautomers and R1 = **coupling** component residue) were prepd. by **coupling** R1H with II or III, prepd. by resp. reaction of R2H or R3H with Z(SO2N3)n [where Z = alkyl or aryl residue, n = 1-3, R4, R6 = O, N(CN), or substituted imino, and R5, R7 = H, heterocyclic residue, optionally substituted alkyl, cycloalkyl,

aryl, aralkyl, or amino]. Thus, benzenesulfonyl hydrazide [80-17-1] was treated with HNO2 to give an emulsion of benzenesulfonyl azide [938-10-3], and barbituric acid [67-52-7] was added to the mixt. to give a suspension of Na **diazobarbiturate** [88638-09-9]. To this suspension 2-cyaniminobarbituric acid [55067-10-2] was added to give Na monocyaniminobarbiturate [88638-10-2] and the suspension was acidified to pH 1 with HCl and heated at 95° to give (IV) [86248-19-3], coloring lacquer and plaster in clear orange shades.

IT **36874-53-0**
(**coupling** of, with **diazobarbituric** acid deriv.)

RN 36874-53-0 HCA

CN 2-Propanone, 1-(2-benzothiazolyl)- (9CI) (CA INDEX NAME)



IC C09B029-036; C09B041-00

CC 41-3 (Dyes, Organic Pigments, Fluorescent Brighteners, and Photographic Sensitizers)

ST azo pigment pyrimidine; benzenesulfonazide **diazo** pyrimidine; cyaniminobarbiturate azo pigment

IT 67-52-7
(conversion of, to **diazo** derivs.)

IT 88638-09-9
(**coupling** of, with barbituric acid derivs.)

IT 119-18-6 1076-38-6 14533-64-3
(**coupling** of, with **diazobarbituric** acid)

IT **36874-53-0** 53815-28-4
(**coupling** of, with **diazobarbituric** acid deriv.)

IT 88638-11-3
(**coupling** of, with imino(cyanimino)barbituric acid)

IT 86248-14-8
(**coupling** of, with **iminodiazobarbituric** acid)

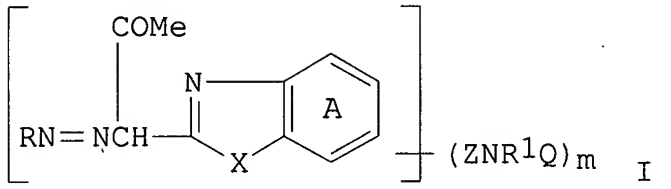
IT 88638-10-2
(**coupling** of, with sodium **diazobarbiturate**)

IT 938-10-3P
(prepn. of, for conversion of barbituric acid derivs. to **diazobarbituric** acid derivs.)

L26 ANSWER 8 OF 13 HCA COPYRIGHT 2007 ACS on STN

100:53196 Reactive azo dyes. Mennicke, Winfried; Fuerstenwerth, Hauke (Bayer A.-G. , Fed. Rep. Ger.). Ger. Offen. DE 3216787 A1 **19831110**, 42 pp. (German). CODEN: GWXXBX. APPLICATION: DE 1982-3216787 19820505.

GI



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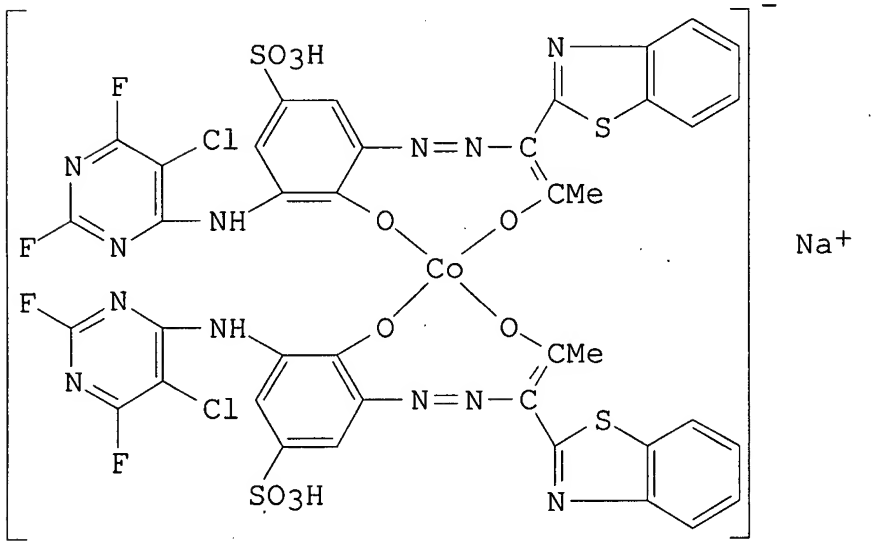


II

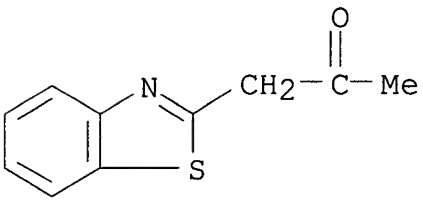
AB Dyes of general structure I (R = **diazo** component residue; X = O, S, NH, substituted NH; Z = direct bond or bridging group; R1 = H, alkyl; Q = fiber-reactive group; m = 1 or 2; ring A may be substituted) and transition metal complexes of I are prepd. I and their complexes are esp. useful as dyes for cotton, polyamide fibers, and wool. Thus, **diazotization** of 2,3,5-HO(AcNH)(HO3S)C6H2NH2 [40306-75-0] and **coupling** with 2-acetonylbenzothiazole [**36874-53-0**], deacetylation, complexing with CoCl2, and reaction with 5-chloro-2,4,6-trifluoropyrimidine [697-83-6] gave II [88475-08-5], a light- and wetfast chocolate brown dye for cotton and wool. Yellow, orange, and other brown dyes were also prepd.

IT **36874-53-0**

(**coupling** of, with **diazotized** (acetamino)aminohydroxybenzenesulfonic acid)

RN 36874-53-0 HCA

CN 2-Propanone, 1-(2-benzothiazolyl)- (9CI) (CA INDEX NAME)



IC C09B062-008; C09B062-012; C09B029-33; D06P001-38

CC 41-3 (Dyes, Organic Pigments, Fluorescent Brighteners, and Photographic Sensitizers)

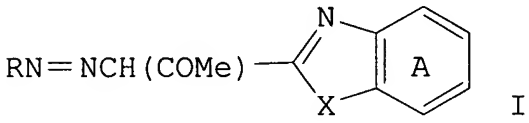
IT 6259-63-8 40306-75-0
(**coupling** of **diazotized**, with acetonylbenzothiazole)

IT **36874-53-0** 88467-77-0
(**coupling** of, with **diazotized** (acetamino)aminohydroxybenzenesulfonic acid)

L26 ANSWER 9 OF 13 HCA COPYRIGHT 2007 ACS on STN

98:217168 Azo dyes and their metal complex dyes. Mennicke, Winfried; Fuerstenwerth, Hauke (Bayer A.-G. , Fed. Rep. Ger.). Ger. Offen. DE 3134944 A1 **19830317**, 24 pp. (German). CODEN: GWXXBX. APPLICATION: DE 1981-3134944 19810903.

GI



I

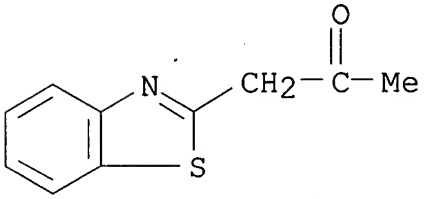
AB Monoazo dyes of general structure I and their metal complexes are prepd., where R represents the residue of a **coupling** component (preferably a benzeneamine or naphthalenamine with a complex-forming substituent), X = O, S, or NR1 (R1 = H, alkyl, aryl, aralkyl, cycloalkyl), and ring A may contain addnl. substituents. The metal complexes (esp. Co) are fast dyes for amide group-contg. materials such as leather, wool, and polyamide fibers. Thus, **diazotization** of 4,3-HO(H2N)C6H3SO2NH2 [98-32-8] and **coupling** with 2-acetonylbenzothiazole [**36874-53-0**] gave I [R = 2,5-HO(H2NSO2)C6H3, X = S] [85935-97-3], which was

then treated with CoSO4 in the presence of Na2CO3 to form the 1:2 Co complex (II) [83249-73-4]. II, a brown powder, dyed wool, polyamide, and leather in reddish light-brown shades with good fastness to light and wet treatment. Other I and I complexes were similarly prepd.

IT **36874-53-0**
(**coupling** of, with **diazotized** aminohydroxybenzenesulfonamide)

RN 36874-53-0 HCA

CN 2-Propanone, 1-(2-benzothiazolyl)- (9CI) (CA INDEX NAME)



IC C09B029-32; C09B045-20; D06P001-04; D06P001-10; D06P003-04

CC 41-3 (Dyes, Organic Pigments, Fluorescent Brighteners, and Photographic Sensitizers)
Section cross-reference(s): 45

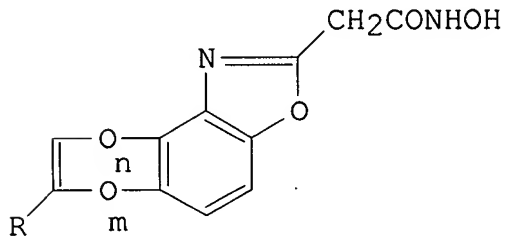
IT 96-93-5 98-32-8
(**coupling** of **diazotized**, with acetonylbenzothiazole)

IT 6635-14-9 **36874-53-0**
(**coupling** of, with **diazotized** aminohydroxybenzenesulfonamide)

L26 ANSWER 10 OF 13 HCA COPYRIGHT 2007 ACS on STN

88:121156 Furobenzoxazolylacethydroxamic acids. Turin, Michel; Guerret, Patrick; Pourrias, Bernard; Ruch, Anne Marie (Delalande S. A., Fr.). Fr. Demande FR 2338041 **19770812**, 16 pp. (French).
CODEN: FRXXBL. APPLICATION: FR 1976-1055 19760116.

GI



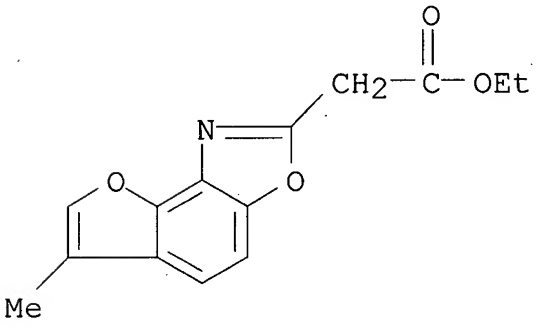
I

AB The title compds. I (n = 0, 1; m = 1, 0; R = C1-4), bronchodilators, were prepd. Thus, **diazo coupling** of aniline and 2-methyl-5-hydroxybenzofuran followed by Na2S2O4 redn. gave 2-methyl-4-amino-5-hydroxy-benzofuran which was cyclocondensed with EtO2CCH2C(:NH)OEt and then amidated with H2NOH.HCl to give I (R = Me, m- = 1, n = 0).(II). II gave 46% relaxation in isolated guinea pig trachea.

IT **65874-34-2P**
(prepn. and amidation of, by hydroxylamine)

RN 65874-34-2 HCA

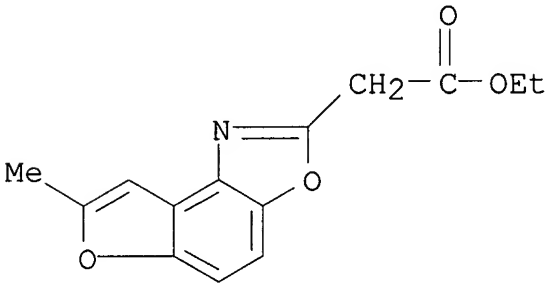
CN Furo[2,3-e]benzoxazole-2-acetic acid, 6-methyl-, ethyl ester (9CI) (CA INDEX NAME)



IT **65874-37-5P**
(prepn. and amidation of, by hydroxylamine)

RN 65874-37-5 HCA

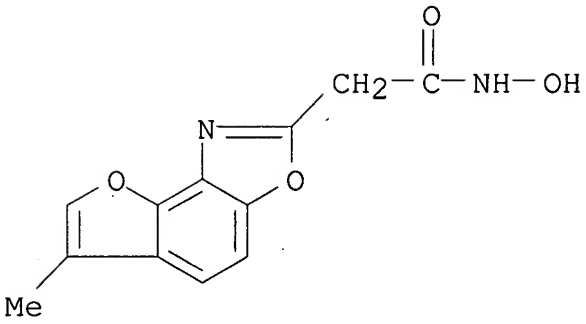
CN Furo[3,2-e]benzoxazole-2-acetic acid, 7-methyl-, ethyl ester (9CI) (CA INDEX NAME)



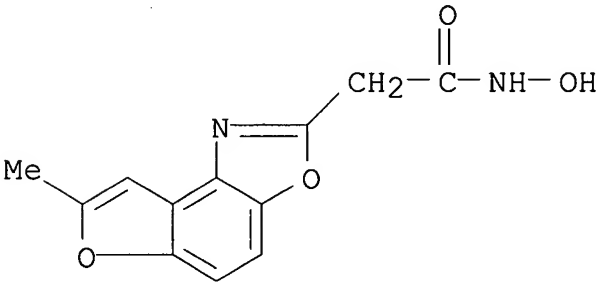
IT **65874-35-3P 65874-38-6P**
(prepn. and bronchodilating activity of)

RN 65874-35-3 HCA

CN Furo[2,3-e]benzoxazole-2-acetamide, N-hydroxy-6-methyl- (9CI) (CA INDEX NAME)



RN 65874-38-6 HCA
CN Furo[3,2-e]benzoxazole-2-acetamide, N-hydroxy-7-methyl- (9CI) (CA INDEX NAME)



IC A61K031-42
CC 28-6 (Heterocyclic Compounds (More Than One Hetero Atom))
IT 6769-56-8
(**coupling** reaction of, with aniline)
IT 62-53-3, reactions
(**coupling** reaction of, with methylhydroxybenzofuran)
IT **65874-34-2P**
(prepn. and amidation of, by hydroxylamine)
IT **65874-37-5P**
(prepn. and amidation of, by hydroxylamine)
IT **65874-35-3P 65874-38-6P**
(prepn. and bronchodilating activity of)

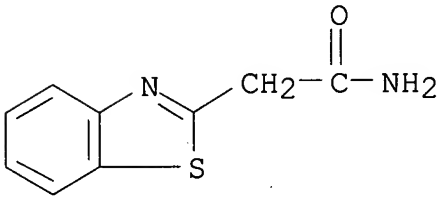
L26 ANSWER 11 OF 13 HCA COPYRIGHT 2007 ACS on STN
80:146965 Azo dyes. Lauer, Dieter; Dehnert, Johannes (BASF A.-G.). Ger. Offen. DE 2232449 **19740110**, 32 pp. (German). CODEN: GWXXBX. APPLICATION: DE 1972-2232449 19720701.
AB **Coupling** of **diazotized** RNH2 (R = substituted phenyl or anthraquinonyl) with 2-(2-benzothiazolyl)acetamides or alkyl (2-benzothiazolyl)acetates gave azo dyes I [R1 = NH2, NH(CH2)3OMe, NHCH2CH2Ph, alkoxy; n = 0 or 1], fast yellow to orange

on polyamide (n = 1) and polyester (n = 0) fibers. Thus, 2,4-O2N(HO3S)C6H3NH2 was **diazotized** and **coupled** with 2-(2-benzothiazolyl)acetamide [**51542-41-7**] (prepd. from o-H2NC6H4SH and NCCH2CONH2) to give azo dye II [51478-86-5], light-and washfast yellow on polycaprolactam. Similarly, **diazotized** 4,2-Cl(O2N)C6H3NH2 was. **Coupled** with 2-(2-benzothiazolyl)-N-phenethylacetamide [**51478-95-6**] to give azo dye I [R = 4,2-Cl(O2N)C6H3, R1 = NHCH2CH2Ph, n = 0] [51478-91-2], fast yellow on polyester fibers. Nine other dyes were prepd.

IT **51542-41-7P**
(prepn. of and **coupling** with **diazotized** aniline derivs.)

RN 51542-41-7 HCA

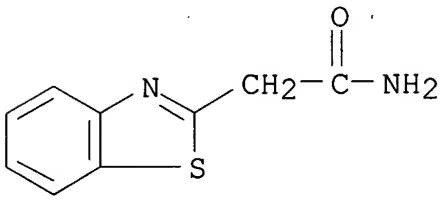
CN 2-Benzothiazoleacetamide (9CI) (CA INDEX NAME)

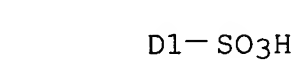


IT **51506-26-4P**
(prepn. of and **coupling** with **diazotized** arom. amines)

RN 51506-26-4 HCA

CN Benzothiazolesulfonic acid, 2-(2-amino-2-oxoethyl)- (9CI) (CA INDEX NAME)



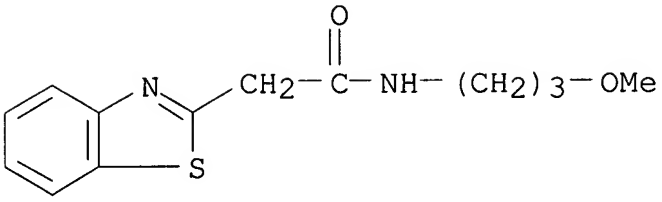


IT **51478-93-4P 51478-95-6P**
(prepn. of and **coupling** with **diazotized** chloronitroaniline)

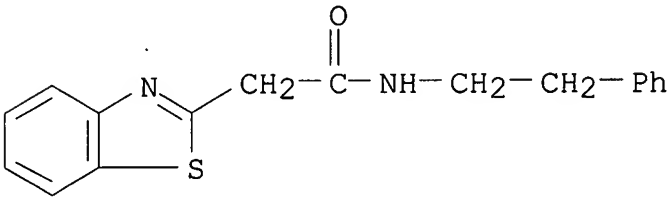
RN 51478-93-4 HCA

CN 2-Benzothiazoleacetamide, N-(3-methoxypropyl)- (9CI) (CA INDEX

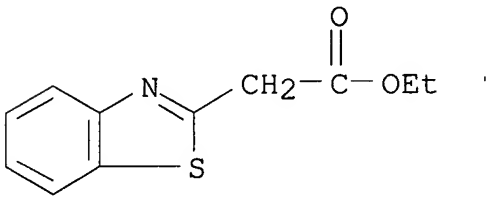
NAME)



RN 51478-95-6 HCA
CN 2-Benzothiazoleacetamide, N-(2-phenylethyl)- (9CI) (CA INDEX NAME)



IT **29182-42-1P**
(prepn. of and **coupling** with **diazotized** nitrosulfoaniline)
RN 29182-42-1 HCA
CN 2-Benzothiazoleacetic acid, ethyl ester (8CI, 9CI) (CA INDEX NAME)



IC C09B
CC 40-4 (Dyes, Fluorescent Whitening Agents, and Photosensitizers)
IT 82-45-1 88-74-4 51478-92-3
(**coupling** of **diazotized**, with (sulfobenzothiazolyl)acetamide)
IT 89-63-4 1817-73-8
(**coupling** of **diazotized**, with benzothiazolylacetamide)
IT 616-84-2
(**coupling** of **diazotized**, with benzothiazolylacetic acid derivs.)
IT **51542-41-7P**
(prepn. of and **coupling** with **diazotized**

aniline derivs.)
IT **51506-26-4P**
(prepn. of and **coupling** with **diazotized** arom. amines)
IT **51478-93-4P 51478-95-6P**
(prepn. of and **coupling** with **diazotized** chloronitroaniline)
IT **29182-42-1P**
(prepn. of and **coupling** with **diazotized** nitrosulfoaniline)

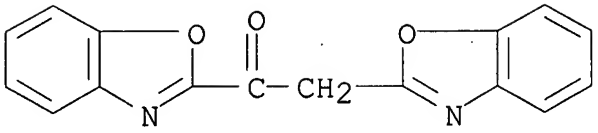
L26 ANSWER 12 OF 13 HCA COPYRIGHT 2007 ACS on STN
55:30422 Original Reference No. 55:5971b-i,5972a-c Azo quinone dyes. Brassel, Jakob; Fasciati, Alfred; Gunst, Raymond; v. Krannichfeldt, Walter (C I B A Ltd.). US 2938024 **19600524** (Unavailable). APPLICATION: US .
GI For diagram(s), see printed CA Issue.
AB A series of dyes of the general formula I was prepd.; in formula I, n = 1-3, R and R' are benzene radicals, X is an amino bridge bound to R' meta to the azo linkage, Y represents the radical of a heterocyclic keto methylene compd. bound to the azo linkage in a vicinal position to a keto group, Z = H or SO3H, and A represents a halogen atom, NH2, or a radical bound to the C atom directly or through an O or N bridge. The new dyes are suitable for dyeing or printing silk, leather, cellulosic, and polyamide and polyurethan fibers. 1-Amino-4-(p-aminophenylamino)anthraquinone-2-sulfonic acid (Ia) 8.18 in H2O 200 contg. NaOH 0.9 and Na2CO3 1.1 treated at 40° with a 25% aq. paste of Na salt of 4,6-dichloro-2-[3-(3-methyl-5-oxo-2-pyrazolin-4-ylazo)-4-sulfophenylamino]triazine (II) 9.8, stirred 0.5 hr. at 35-40° and 0.5 hr. at 45-50°, heated to 70°, and filtered, and the residue washed with 2% aq. NaCl 150 parts yielded a dark powder (III), green in H2O; it dyes wool from a neutral or weakly acidic bath clear green tints. III refluxed 6 hrs. with H2O 1000 and PhNH2 3.8, dild. with satd. aq. NaCl 120 parts, stirred 15 min., and filtered hot yielded a dark powder (IV), green in H2O; it dyes clear green tints. Cyanuric chloride (V) 18.5 in iced H2O 400 and 3-methyl-4-(5-amino-2-sulfophenylazo)-5-pyrazolone (VI) 29.7 [obtained by **coupling** **diazotized** 3,4-H2N(HO3S)C6H3NHAc (VII) with 3-methyl-5-pyrazolone] in H2O 400 treated dropwise with Na2CO3 10.6 in H2O 100 parts during 3 hrs. at 2-5° yielded the Na salt of II. The condensation product 39.3 of V, PhNH2 and 2,4-(H2N)2C6H3SO3H (1:1:1 mole) in H2O 300 and 30% HCl 25 **diazotized** at about 10-15° with 4N aq. NaNO2 25, treated with NaOAc.3H2O 30 and then slowly at 10-15° with 1-(o-chlorophenyl)-3-methyl-5-pyrazolone 21.5 in N NaOH 104, and filtered, the residue stirred with H2O 1000 at 60°, treated with Ia 40.9 in H2O 500 parts at 60°, heated to 90-5°,

neutralized with dil. aq. Na2CO3, cooled to 65°, salted with NaCl, and filtered gave a green powder which produced on wool yellowish green tints. VII 23.0 **diazotized** and **coupled** with 1-(2-methyl-6-chlorophenyl)-3-methyl-5-pyrazolone 20.8, the resulting dye sapond. with aq. NaOH at 90-5°, neutralized at 50°, and filtered, the resulting amino azo dye 45 in H2O 650 added at 60-70° to V 18.4 in ice 200 and H2O 50 with stirring, stirred 4 hrs. at 10-15° while being neutralized with N NaOH, the mixt. treated with a neutralized soln. of Ia 40.9 in H2O 500, stirred 12 hrs. at 40° while being neutralized with N NaOH, treated with PhNH2 18.0, stirred 2.5 hrs. at 90-5°, cooled to 80°, treated with NaCl about 240 parts, stirred, and filtered gave a green dye. The 6-PhNH analog 5.2 of II in H2O 200 and Ia 4.09 and NaOH 0.4 in H2O 200 refluxed 24 hrs. while being neutralized with dil. aq. Na2CO3, treated with satd. aq. NaCl 60 parts, and filtered at 50-5° gave a dark powder, green, in H2O; it dyes clear green tints. V 18.5 in ice 600 and H2O 200 treated with neutralized 2,4-(H2N)2C6H3SO3H 18.8 in H2O 400 while being kept at pH 6.5 by the continuous addn. of N NaOH 100 parts, kept 15 min. at 0°, adjusted with aq. NaOH to pH 8.5, treated with 4N aq. NaNO2 25, added during 15 min. into a soln. 60 contg. 30% 1-C10H7SO3H, stirred 2 hrs. at 20°, treated with barbituric acid 12.8 in H2O 400 and 10% aq. Na2CO3 200, salted with NaCl, and filtered, the residue in H2O 1000 treated with di-Na salt of 1-amino-4-(4-amino-3-sulfophenylamino)-2-sulfonic acid (VIII) in H2O 500 at 60°, stirred 24 hrs. at 40-5° at pH 6.5, alkalinized with Na2CO3 5 parts, and salted gave a dye which produced on cellulose fibers from an alk. bath contg. a salt, alkali-fast, bluish green tints. The di-Na salt (0.02 mole) of VIII in cold H2O 400 cc. treated at 0° with V 3.8 g. in Me2CO 20 cc., stirred 20-30 min. at 0-5° while being neutralized with dil. NaOH, neutralized VI 0.02 mole in H2O 400 cc. added, stirred about 7 hrs. at 35-40° while being neutralized continuously with dil. NaOH, and salted gave a dark powder, green in H2O; it dyes fast, green shades; it can also be obtained by treating VI 1 with V 1 and then condensing the product with VIII 1 mole. Examples for the dyeing of wool and cotton with some of the dyes are given.

IT **92792-24-0P**, Ketone, 2-benzoxazolyl 2-benzoxazolylmethyl (prepn. of)

RN 92792-24-0 HCA

CN Ketone, 2-benzoxazolyl 2-benzoxazolylmethyl (6CI, 7CI) (CA INDEX NAME)



CC 25 (Dyes and Textiles)

IT **92792-24-0P**, Ketone, 2-benzoxazolyl 2-benzoxazolylmethyl
106322-30-9P, 2-Anthraquinonesulfonic acid, 1-amino-4-[4-[[4-chloro-6-[3-(3-methyl-5-oxo-2-pyrazolin-4-ylazo)-4-sulfoanilino]-s-triazin-2-yl]amino]-3-sulfoanilino]- 106480-97-1P, 2-Anthraquinonesulfonic acid, 1-amino-4-[p-[[4-anilino-6-[3-[1-(6-chloro-o-tolyl)-3-methyl-5-oxo-2-pyrazolin-4-ylazo]-4-sulfonanilino]-s-triazin-2-yl]amino]anilino]- 106570-36-9P, 2-Anthraquinonesulfonic acid, 1-amino-4-[p-[[4-anilino-6-[3-[1-(o-chlorophenyl)-3-methyl-5-oxo-2-pyrazolin-4-ylazo]-4-sulfoanilino]-s-triazin-2-yl]amino]anilino]- 106784-30-9P, 2-Anthraquinonesulfonic acid, 1-amino-4-[p-[[4-anilino-6-[3-(3-methyl-5-oxo-2-pyrazolin-4-ylazo)-4-sulfoanilino]-s-triazin-2-yl]amino]anilino]- 108747-95-1P, p-Benzenedisulfonic acid, 2-[4-[5-[[4-[p-[(4-amino-3-sulfo-1-anthraquinonyl)amino]phenyl]-6-chloro-s-triazin-2-yl]amino]-2-sulfophenylazo]-3-methyl-5-oxo-2-pyrazolin-1-yl]- 109512-17-6P, 2-Anthraquinonesulfonic acid, 1-amino-4-[p-[[4-[3-[1-(o-chlorophenyl)-3-methyl-5-oxo-2-pyrazolin-4-ylazo]-4-sulfoanilino]-6-(cyclohexylmethylamino)-s-triazin-2-yl]amino]anilino]- 117867-09-1P, 2-Anthraquinonesulfonic acid, 1-amino-4-[4-[[4-chloro-6-[3-(hexahydro-2,4,6-trioxo-5-pyrimidinylazo)-4-sulfoanilino]-s-triazin-2-yl]amino]-3-sulfoanilino]- 117879-24-0P, 2-Anthraquinonesulfonic acid, 1-amino-4-[p-[[4-chloro-6-[3-(3-methyl-5-oxo-2-pyrazolin-4-ylazo)-4-sulfoanilino]-s-triazin-2-yl]amino]anilino]-
(prepn. of)

L26 ANSWER 13 OF 13 HCA COPYRIGHT 2007 ACS on STN

49:15979 Original Reference No. 49:3153h-i,3154a-i,3155a-i,3156a-i,3157a-i,3158a-b Attempted synthesis of penicillins. Bachmann, W. E.; Cronyn, M. W. (Univ. of Michigan, Ann Arbor). Chem. of Penicillin (H. T. Clarke, et al.)(Princeton Univ. Press) 849-91 (Unavailable) **1949.**

GI For diagram(s), see printed CA Issue.

AB In the studies of the synthesis of penicillin, many of the procedures which involved a cyclization in the final step were theor. capable of yielding either the β-lactam or oxazolone-thiazolidine structures. Tests for antibiotic activity were employed as criteria of the potential usefulness of a reaction but no activity greater than 1-2 units per mg. (0.1% activity) was ever obsd. Representative attempts to activate penicilloic acids are reported. In these numerous azlactonizing expts. the agents

used included Ac2O, Ac2O in pyridine, acid chlorides, phosphorus trihalides, POCl3, PCl5, azlactones, and aroyl azides. Various dehydrating agents and adsorbents such as CaCl2, CuSO4, P2O5, Al2O3, silica gel, Nuchar, etc., were also tried. Control expts. to det. the stability of benzylpenicillin or its β-ester under these operating conditions were performed. Benzylpenicilloic acid, PhCH2CONHCR(CO2H)CH.S.CMe2.CH(CO2H).NH (I, R = H) (Ia) and its esters in the form of racemic mixts. or of optically active isomers and various homologs and analogs were employed. These expts. are classified and tabulated. The few products isolated and characterized proved to be penicillenates formed by cleavage of the thiazolidine ring after azlactonization, the extent of which was detd. by difference from the yield of α-benzylamide. To prevent formation of penicillenates, it was planned to use 6-alkylpenicilloic acid derivs. (I, R = alkyl), but no such compds. were available. Similar blocking attempts by utilizing the benzyl thioamide (instead of the amide) and α-thio ester derivs. of penicilloic acids failed to yield antibiotic active compds. Procedures designed particularly to produce compds. with the β-lactam structure included the action of Grignard reagents on α-alkyl and dialkylpenicilloates. Treatment of benzylpenicilloic acid α-ester with BuMgBr, carbonation, and pyrolysis of the product at 210-50° produced inactive material. Although β-methyl-D-γ-benzylpenicilloate (Ib) treated with PBr3 in dioxane gave a product with a 5.6 μ bond in the IR, characteristic of the β-lactam CO group, attempts to isolate the active material by treatment of the mixt. with CH2N2 or bases were unsuccessful. Various attempts to form the β-lactam structure by elimination of CO2, CO, N, etc., from 5-8 membered rings produced by the closure of suitably substituted penicilloates were unsuccessful, as were efforts based on the elimination of the elements of BzH, NaBr, etc., from similar compds. Prepn. of active compds. was attempted from α-amides, α-hydrazides, and N4-acylpenicilloates. A mixt. of benzylpenicilloic acid α-amide (Ic) (401 mg.) and 156 mg. BF3.Et2O complex in 10 mL. dioxane was heated to 100° without sepn. of the BF3.NH3 complex, indicating no reaction. Similarly, cyclization of the HCl salt by heating alone or in solvents could not be accomplished Attempts to form a triazine and to arrive at the β-lactam by thermal decompn. were made by converting Ic to the α-amido-N-nitroso compd., transformed by treatment with NaOH in dioxane or KOH in MeOH to a compd., m. 133-4°, [α]D23 16° (c 0.49%, EtOH). No antibiotic activity resulted from the thermal treatment of this "triazine" nor was any significantly active material obtained from the product of the nitrosation of the α-hydrazides of the β-esters of benzyl- and phenylpenicilloic acids. Dropwise treatment of 36.6 g. α-Me D-benzylpenicilloate (II) in 50 mL. CHCl3 contg. 8 mL.

pyridine with 10 mL. Me2CHCOCl with stirring at room temp. yielded 45 g. α-Me N-isobutyryl-D-benzylpenicilloate; α,β-di-Me ester, m. 123-4°, hydrolyzed by NaOH in aq. MeOH to the β-Me ester, m. 206-7°. Formylation of II produced an amorphous N4-formyl deriv. No active material was obtained on pyrolysis of these N4-derivs. Pyrolysis of α-Et β-Me N4-acetyl-N8-methyl-9-phenylpenicilloate gave a compd. with slight antibiotic activity, but none was obtained by the pyrolysis of the monoester or the corresponding N4-isobutyryl compds. The prepn. of benzylpenicillin with the β-lactam structure was attempted by cyclization of di-Me N-carbophenoxy-D-benzylpenicilloate (III) by the Dieckmann procedure. III (125 mg.) in 3 mL. Et2O and (ClCH2)2 was added with stirring to 2 equivs. Me2CHMgI in 3 mL. Et2O. Decompn. of the gummy complex with 2N H2SO4 gave 80% unchanged III and a small yield of gum which, on sapon. in pyridine, showed no antibiotic activity. Similarly, the di-Me ester of N4-methoxalylbenzylpenicilloic acid failed to cyclize on treatment with NaOMe, Ph3CNa, BF3, or Me2CHMgBr. Possible prepn. of compds. with the β-lactam structure from cyclization of N-(N-phenylacetyl- β,β-diethoxyalanyl)penicillamine (IV) was early envisaged. In the prepn. of IV, direct formylation of the Et ester of phenaceturylpenicillamine by the action of HCO2Et and Na was unsuccessful. Addn. of 0.523 g. NaNO2 in H2O to 4 g. benzylpenaldic acid di-Et acetal hydrazide in 2N HCl at 0° gave the gummy azide, which was **coupled** with 1.374 g. penicillamine (V) HCl salt by stirring with 0.80 g. Na2CO3 and 0.637 g. NaHCO3 in 15 mL. H2O 2 h. and recrystg. from aq. EtOH to give colorless needles of IV, m. 67°. Similar condensation of the azide from benzylpenaldic acid di-Me acetal hydrazide, m. 180° with D-V.HCl gave N-(N-phenylacetyl-β,β-dimethoxyalanyl)-D-penicillamine (VI), m. 115-16°, [α]D25 24° (c 1.0, MeOH); Me ester, m. 96°, [α]D25 36° (c 0.1, MeOH). Condensation of the azide from α-phenylacetamide-β,β-dimethoxypropionic acid hydrazide with V Me ester produced a "urea," m. 122-3°, [α]D25 -27° (c 1,0, MeOH) which evolved H2S on heating at 100° in Ac2O to yield an inactive compd., m. 151-2°. VI showed considerable thermal stability and fusion alone at 150° or with pyridine-HCl at 140°; these fusions gave material with 0.2-0.5 unit activity per mg. Ring closures of the ester were attempted. Treatment of 2-phenyl-4-ethoxymethylene-5-oxazolone in 2N HCl with abs. EtOH 2 days at room temp. produced N-benzyl-β,β-diethoxyalanine Et ester, m. 48°; hydrazide (VII), m. 154-5°, converted by warming at 100° for 1 h. with 2N HCl and EtOH to 2-benzoylamino-3-pyrazolone, m. 200-1°. Condensation of the azide from VII with V.HCl gave N-(N-benzoyl-β,β-diethoxyalanyl)penicillamine (VIII), m. 150°; Me ester, m.

90-1°, not cyclized by cold Ac2O nor by hot pyridine or Ac2O. Condensation of 20 g. of D-V Me ester-HCl and 22 g. 2-benzyl-4-methoxymethylene-5-oxazolone in 100 mL. pyridine by the addn. of 100 mL. MeOH gave a compd. (IX), m. 140-1°, [α]D22 100.7° (c 1.45, MeOH) and N-(α-phenylacetylamino-β-methoxyacrylyl)penicillamine Me ester, m. 108.9°, transformed by refluxing with Et2O to IX, which gave a neg. test for sulfhydryl group, and was hydrogenolyzed over Raney Ni to β-methoxy-N-phenylacetylalanyl-D-valine Me ester, m. 86-7°, identical with a synthetic prepn. Heating IX 2 h. in pyridine at 78-80° or in xylene 16 h. with a trace of Et2NH gave material with no antibiotic activity. Attempts to effect cyclization of penicillenates with the formation of a thiazolidine ring were fruitless. No antibiotic activity resulted when Me benzyl- or amylpenicillenates were kept in pyridine at room temp. 1 day or on treating the crude penicillenates from the condensation of V and 2-benzyl-4-alkoxy-5-oxazolones with toluene alone or with ascaridole, BzO2H, N-ethylpiperidine, Ib, or by treatment with pyridine and Cu(OAc)2. Various attempts to isomerize penillic to penicillenic acids by UV radiation, AlCl3 in dioxane, Al(OBu-tert)3 in dioxane, pyridine with ascaridole and with BzO2H, various acids in (ClCH2)2, BF3, and PhNCO failed to bring about the reversal. Treatment of the oxazole-thiazolidine, N:CPh.O.CCl:CCH.S.CMe2.CH(CO2H).NH, with dry pyridine at 60° for 5 h. yielded antibiotically active material (0.25-0.5 units per mg.), quickly inactivated by the action of penicillinase. Condensation of 2-benzyl-4-oxazolecarbonylchloride and V Me ester gave an acylpenicillamine deriv. Portions (3 mL.) of a mixt. of 468 mg. of Me D-5,5-dimethyl-2-thiazoline-4-carboxylate (X) and 444 mg. 2-benzyl-5-oxazolone in 9 mL. toluene were refluxed, and heated at 100° and at 65-70° for 10-min. periods. Samples of the reaction products were sapond. and assayed in vitro but showed no activity. No biol. activity was found in products obtained from the condensation of equimolar quantities of 2-phenyl- or 2-amyl-5-ethoxyoxazole with X. To provide a necessary acylaminoketene for reaction with X to produce a compd. with β-lactam structure, 2.64 mL. PhCH2COCl was added to a suspension of Hg2(NCO)2 in 15 mL. dry benzene and the filtered soln. was satd. with dry HCl to give presumably PhCH2CONHCOCl, m. 105-8° (phenylacetylurea, m. 209-10°). Treatment with excess CH2N2 gave presumably PhCH2CONHCOCHN2, which was rearranged with Ag2O to PhCH2CONHCHC:O in the presence of X to yield biol. active but not reproducible products. A large no. of investigations were concerned with the prepn. of "dehydropenicillins" of the structure N:C(CH2Ph).O.CO.CHC:N.CH(CO2H).CMe2.S, or N:C(CH2Ph).O.CO.C:C.NH.CH(CO2H).CMe2.S (XI), which would give the oxazolone-thiazoline structure on redn. Refluxing 15 g. NaH4.H2O with 20.7 g. PhCH2CONHCH2CO2Me in 50 mL. MeOH 1 h. and recrystg. the

product from Me2CHOH yielded phenaceturyl hydrazide, m. 130-2°, converted to the azide, m. 85-6°, which was condensed with V.HCl to crude D-N-phenaceturylpenicillamine, m. 137-40°. Cyclization by standing for 5 days in satd. ethereal HCl gave a product whose anal. corresponded to that of dehydrobenzylpenilloic acid-HCl (XII). Simultaneous addn. of 125 g. PhCH2COCl and 32 g. NaOH in H2O below 0° to 100 g. of H2NCH2CN.H2SO4 in 500 mL. H2O contg. 52 g. NaOH yielded 70 g. phenylacetamidoacetonitrile, m. 93, converted by treatment with dry HCl at 0° in dioxane and MeOH to phenylacetamidoacetimino Me ether-HCl, m. 158° (Et ether-HCl, m. 165°), yielding with excess Na2CO3 in Et2O the corresponding ethers (Me, m. 80-1°; Et, m. 91-2.5°). Condensation of either of these ethers with V Me ester-HCl gave XII Me ester, b0.1 180-90°, reduced in Et2O over Al-Hg to Me benzylpenilloate (HCl salt, m. 85-95°), cleaved by HgCl2 to benzylpenilloaldehyde, identified by the 2,4-dinitrophenylhydrazone, m. 195-8°. A mixt. of 1.8 g. H2NCH(CO2Et)2 in 25 mL. Et2O and 1.5 g. Na2CO3 in 10 mL. H2O was shaken and 1.5 g. PhCH2COCl was added dropwise; warming to complete reaction and sepg. the Et2O layer gave di-Et phenylacetamidomalonate, m. 67-8°. Treatment of 1.08 g. of this ester in 5 mL. EtOH contg. 0.21 g. KOH, evapn. to dryness, soln. in H2O, acidification and recrystn. from CHCl3-petr. ether produced mono-Et phenylacetamidomalonate, m. 104-5°. This half-ester was converted to the hydrazide, m. 143-5°, and then to the colorless cryst. azide, which was filtered off and added to V.HCl in aq. Na2CO3. After 15 min. the mixt. was acidified with HCl to yield N-(N-phenylacetyl-α-carboxyglycyl)penicillamine, m. 152-3°. The compd. appeared to react with ethereal HCl but no cryst. products were isolated. Similarly, the monoazide of benzoylaminomalonic acid was **coupled** with V and its Me ester without prodn. of cryst. material. No definite products were obtained from N-phenaceturylpenicillamine Me ester and CH(OEt)3, HCSNH2 or CHCl3. Another approach employed 2-benzyl-4-carbethoxy-5-oxazolone (XIII). Phenylacetamidomalonic acid ester hemihydrate (1 g.) was warmed on the steam bath 30 min. with 10 mL. Ac2O, freed from excess reagent in vacuo, and distd. in vacuo at 50-60° gave PhCH2 CONHCH2CO2Et, m. 79-80°. XIII reacted readily with PhNH2 and p-H2NC6H4Me to produce phenylacetamidomalonanilic Et ester, m. 156°, and the corresponding toluidide Et ester, m. 157-8°. Addn. of 1 g. crude XIII to 500 mg. of cysteine Me ester in 15 mL. benzene and 5 mL. AcOEt and recovery of the residue from Et2O gave N-(N-phenylacetyl-α-carbethoxyglycyl)cysteine Me ester, m. 106-20°. Similarly, allowing a mixt. of XIII and V Me ester to stand in Et2O overnight, extg. with 2N HCl and aq. Na2CO3, concg. the Et2O ext. and recrystg. the residue from CHCl3-petr. ether gave N-(N-phenylacetyl-α-

carbethoxyglycyl)penicillamine Me ester, m. 128-9°, not convertible into the thiazolidine by ethereal HCl. The hippuryl analog similarly failed to cyclize in methanolic HCl. The desired "dehydropenicillin" was successfully synthesized from 2-carbethoxymethyl-4-carbomethoxy-5,5-dimethylthiazoline (XIV); this with **benzenediazonium** chloride gave the phenylazo deriv., m. 120°. V Me ester (3.2 g.) in 5 mL. CH2(CO2Et)2 was added dropwise to 10 mL. C2H(CO2Et)2 at 175°. After distn. in vacuo the residual oil was distd. at high vacuum, yielding 2.5 g. XIV, b0.018 156°, m. 109-11°. XIV (10.2 g.) in 75 mL. EtOH and 75 mL. 2N HCl was treated dropwise with stirring with 5.0 g. NaNO2 in 20 mL. H2O at 0°. After 15 min., the mixt. was dild. with H2O to yield 2-isonitrosocarbethoxymethyl-4-carbomethoxy-5,5-dimethylthiazoline, m. 141°. Warming 0.4 g. of nitroso compd. in 8 mL. 2N NH4OH for 5-10 min. on the steam bath with 1.2 g. Na2S2O4 in 5 mL. H2O gave 2-aminocarbethoxymethyl-4-carbomethoxy-5,5-dimethylthiazoline (XV).HCl, m. 163-7°. Phenylacetylation of 1.8 g. XV oxalate by stirring for 2.5 h. with 25 mL. Et2O, 1.5 g. NaHCO3, and 0.8 g. Ph2CH2COCl yielded 4-carbomethoxy-5,5-dimethyl-2-phenylacetamidocarbethoxymethylthiazoline (XVI), m. 136-7° (α-Et β-Me "benzyldehydropenicilloate"). Treatment of 0.6 g. XVI in 10 mL. EtOH with 3.06 mL. 0.51 N NaOH for 1 h. and acidification of the filtrate with 1 equiv. 0.5N HCl at 0° produced 4-carboxy-5,5-dimethyl-2-phenylacetamidocarbethoxymethylthiazoline, m. 120-4° (decompn.); morpholine salt, m. 173°, by preferential hydrolysis of the β-ester group. Refluxing 15 g. XVI gently with 220 mL. CHCl3 and 8.5 g. PCl5 50 min., allowing to stand at room temp. several hrs., washing with aq. NaHCO3, chromatographing over Al2O3, and recrystg. from CHCl3-petr. ether yielded 7.5 g. "thiazolineoxazolone" (XVII, R = PhCH2), m. 118-19°. The same compd. was produced from the corresponding α-benzyl β-Me ester (XVIII) by loss of the elements of PhCH2OH. This remarkable formation of oxazolones rather than oxazoles by ring formation suggests that the precursors may have the structure RCONHC(CO2R'):C.S.CMe2.CH(CO2R").NH, and yield by loss of the elements of R'OH compds. such as XVII. The p-nitrobenzamide analogs of XVII and XVIII and the corresponding compds. of the caproamido series were similarly prepd., providing the following compds.: 4-carbomethoxy-5,5-dimethyl-2-p-nitrobenzamidocarbethoxymethylthiazoline (XIX), m. 173°; 4-carboxy acid, m. 112°, remethylated to XIX. Shaking 4.23 g. XIX with 39.4 g. 0.51N NaOH 15 h., acidifying the filtrate at 0°, and purifn. through the Pb salt by decompn. with H2S gave 4-carboxy-5,5-dimethyl-2-p-nitrobenzamidomethylthiazoline, m. 110° (softening). Refluxing 0.8 g. XIX in 15 mL. dry CHCl3 with 1 g. PCl5 1 h. and chromatographing over Al2O3 yielded yellow prisms of 4-(4-carbomethoxy-5,5-dimethylthiazolin-2-yl)-5-ethoxy-2-(p-nitrophenyl)oxazole, m. 205°. Cyclization of XIX by

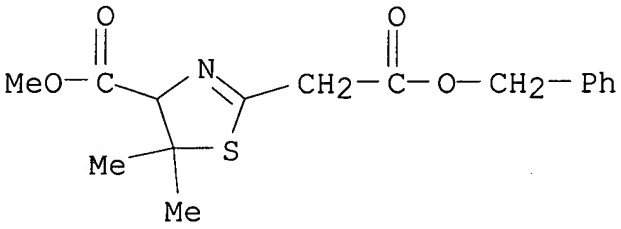
refluxing in CHCl3 over PCl5, chromatographing the washed CHCl3 soln. over Al2O3, and recovering material from the upper part of the column gave XVII (R=p-O2NC6H4) (XX), m. 265°. Caproylation of XV oxalate yielded 4-carbomethoxy-5,5-dimethyl-2-caproamidocarbethoxymethylthiazoline (XXI), m. 104-5°; 4-carboxy acid, m. 149-50°. Cyclization of XXI produced XVII (R = Am) (XXII), m. 87-8°. Heating 30 mL. NCCH2CO2Et with 150 mL. PhCH2OH at 194-200° for 3 h. and removal of the residual PhCH2OH at 100° and 18 min. yielded 34 g. NCCH2CO2CH2Ph, b0.5 141°, nD19 1.5206. A mixt. of 17.5 g. ester and 4.6 g. anhyd. EtOH was treated with 3.8 g. dry HCl overnight, yielding 23.5 g. carbobenzyloxyacetimino Et ether-HCl, m. 89° (effervescence). Condensation of 5.1 g. HCl salt with 4.0 g. V Me ester, 2.5 g. AcOK, 5 mL. H2O, and 5 mL. Et2O by shaking together 2 h. yielded 3 g. 4-carbomethoxy-5,5-dimethyl-2-carbobenzyloxymethylthiazoline, m. 78°, converted to the oily 2-isonitroso deriv., reduced over HgAl in EtOH, and crystd. Me2CO-Et2O in Et2O to give 12.8 g. 4-carbomethoxy-5,5-dimethyl-2-aminocarbobenzyloxymethylthiazoline; oxalate (XXIII), m. 120-1°, phenylacetylated to XVIII, m. 132-3°; caproylated to the 2-caproamido deriv. (XXIV), m. 115°, and p-nitrobenzoylated to the 2-p-nitrobenzamido compd. (XXV), m. 182-3°. XVIII was sapond. to the 4-carboxy acid (XVIIIa), m. 153-4°. Cyclization of XVIII, XXIV, and XXV produced the "thiazoline-oxazolones" XXII, XX, and XVII. Cyclization of XVIIIa gave a "thiazoline-oxazolone" acid (XXVI), m. 190° (hemihydrate, m. 122-3° (decompn.); HCl salt, m. 165° (decompn.)) also obtained by hydrolysis of XVII. Methylation of XXVI with excess CH2N2 in ether gave the stereoisomeric N4-Me derivs. of the β-Me esters, m. 151-2° and 110-111°. Many attempts were made without success to reduce XXV and the caproamido analog XXII and their Me esters to the penicillins or their esters. No appreciable biol. activity developed and vigorous redn. led by breakdown to unidentified products. In another procedure 13.3 g. PhCHClCOCl was added dropwise with cooling and stirring to 12 g. β,β-diethoxyalanine in 150 mL. N NaOH. After extn. with CHCl3, the aq. layer was acidified with 2N H2SO4, the oily product was taken up in Et2O, dried, and heated with excess CH2N2 in Et2O. Distn. in high vacuum gave 12.6 g. pure Me α-chlorobenzylpenaldate di-Et acetal, m. 72-4°; 2,4-dinitrophenylhydrazone, m. 153-4°. Heating 3.3 g. acetal in 7 mL. glacial AcOH with 1.5 g. V.HCl.H2O 30 min. and pptn. with 150 mL. dry Et2O gave 2.83 g. α-Me DL-(α-chlorobenzyl)penicilloate-HCl, sintering at 95°, decomp. at 180°. Treatment of 10.52 g. HCl salt with 69.3 mL. N NaOH overnight and neutralization at 0° with 46.2 mL. N HCl yielded 5.2 g. DL-chlorobenzylpenicilloic acid, m. 85-90° (decompn.), converted by shaking with 10.8 g. pyridine

and 35.2 mL. Ac2O to "benzyldehydropenicillin," m. 90-5° (decompn.), with the probable structure PhCH:C.O.CO.CMe:N. All attempts at redn. failed. None of the expts. performed yielded penicillin. No active products were obtained from the action of phenylketene di-Me acetal on D-4-carbomethoxy-5,5-dimethyl-α-amino-2-thiazolidineacetic acid (XXVII) or of PhCCl3 on the Na salt of XXVII in the presence of NaHCO3, NEt3, or pyridine. The reaction of COCl2 with XXVII gave a bicyclic product (XXVIII), m. 168-9° (decompn.), [α]D23 215° (EtOH), which was heated with PhCH2MgCl in the hope that the Grignard product would undergo cyclization to penicillin Me ester. However, no activity was found in the reaction product. Since XXVIII was shown to have an active H atom, the use of MeCH2CH:CHMgBr was later proposed (C.A. 39, 2968.2).

IT **875237-80-2**, 2-Thiazoline-2-acetic acid, 4-carboxy-5,5-dimethyl-, 2-benzyl 4-Me ester (and isomer)

RN 875237-80-2 HCA

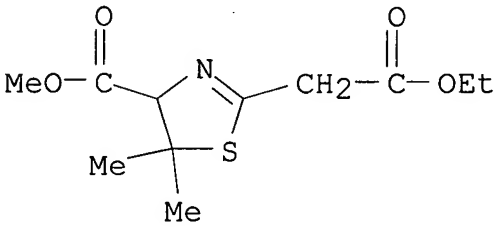
CN 2-Thiazoline-2-acetic acid, 4-carboxy-5,5-dimethyl-, 2-benzyl 4-Me ester (5CI) (CA INDEX NAME)



IT **721457-39-2P**, 2-Thiazoline-2-acetic acid, 4-carboxy-5,5-dimethyl-, 2-ethyl 4-methyl ester (prepn. of)

RN 721457-39-2 HCA

CN 2-Thiazoline-2-acetic acid, 4-carboxy-5,5-dimethyl-, 2-ethyl 4-methyl ester (5CI) (CA INDEX NAME)



CC 10 (Organic Chemistry)

IT **875237-80-2**, 2-Thiazoline-2-acetic acid,

4-carboxy-5,5-dimethyl-, 2-benzyl 4-Me ester
(and isomer)